

L Number	Hits	Search Text	DB	Time stamp
1	50	((("0245155") or ("5780406") or ("5705089") or ("5334332") or ("5466389") or ("5308745") or ("4111767") or ("6503633") or ("5935920") or ("4617251") or ("6465368") or ("6495264") or ("5417877") or ("6495506") or ("Re30796") or ("5279771") or ("4239661") or ("5750482") or ("6339054") or ("5614027") or ("4786578") or ("4067690") or ("3954643") or ("6479374") or ("6110451") or ("6495573") or ("6500599") or ("6506937") or ("6489080") or ("6475700") or ("4824763") or ("6506537") or ("4549968") or ("6506318") or ("6492086") or ("6506759") or ("6482567") or ("6479211") or ("5646240") or ("6479039") or ("5744504") or ("6491970") or ("6482848") or ("6506419") or ("6498163") or ("6506738") or ("4350606") or ("6458849") or ("3582401") or ("3160539")).PN.	USPAT	2003/01/15 13:01
2	50	((("6245155") or ("5780406") or ("5705089") or ("5334332") or ("5466389") or ("5308745") or ("4111767") or ("6503633") or ("5935920") or ("4617251") or ("6465368") or ("6495264") or ("5417877") or ("6495506") or ("Re30796") or ("5279771") or ("4239661") or ("5750482") or ("6339054") or ("5614027") or ("4786578") or ("4067690") or ("3954643") or ("6479374") or ("6110451") or ("6495573") or ("6500599") or ("6506937") or ("6489080") or ("6475700") or ("4824763") or ("6506537") or ("4549968") or ("6506318") or ("6492086") or ("6506759") or ("6482567") or ("6479211") or ("5646240") or ("6479039") or ("5744504") or ("6491970") or ("6482848") or ("6506419") or ("6498163") or ("6506738") or ("4350606") or ("6458849") or ("3582401") or ("3160539")).PN.	USPAT	2003/01/15 13:01
3	41	((("6245155") or ("5780406") or ("5705089") or ("5334332") or ("5466389") or ("5308745") or ("4111767") or ("6503633") or ("5935920") or ("4617251") or ("6465368") or ("6495264") or ("5417877") or ("6495506") or ("Re30796") or ("5279771") or ("4239661") or ("5750482") or ("6339054") or ("5614027") or ("4786578") or ("4067690") or ("3954643") or ("6479374") or ("6110451") or ("6495573") or ("6500599") or ("6506937") or ("6489080") or ("6475700") or ("4824763") or ("6506537") or ("4549968") or ("6506318") or ("6492086") or ("6506759") or ("6482567") or ("6479211") or ("5646240") or ("6479039") or ("5744504") or ("6491970") or ("6482848") or ("6506419") or ("6498163") or ("6506738") or ("4350606") or ("6458849") or ("3582401") or ("3160539")).PN.) and (ammonium or fluoride or sulfoxide or dmsol or sulfone or chelating or chelate or lactate)	USPAT; US-PGPUB; EPO; JPO; DERWENT; IBM_TDB	2003/01/15 13:06
4	7	((("6245155") or ("5780406") or ("5705089") or ("5334332") or ("5466389") or ("5308745") or ("4111767") or ("6503633") or ("5935920") or ("4617251") or ("6465368") or ("6495264") or ("5417877") or ("6495506") or ("Re30796") or ("5279771") or ("4239661") or ("5750482") or ("6339054") or ("5614027") or ("4786578") or ("4067690") or ("3954643") or ("6479374") or ("6110451") or ("6495573") or ("6500599") or ("6506937") or ("6489080") or ("6475700") or ("4824763") or ("6506537") or ("4549968") or ("6506318") or ("6492086") or ("6506759") or ("6482567") or ("6479211") or ("5646240") or ("6479039") or ("5744504") or ("6491970") or ("6482848") or ("6506419") or ("6498163") or ("6506738") or ("4350606") or ("6458849") or ("3582401") or ("3160539")).PN.) and (ammonium or fluoride or sulfoxide or dmsol or sulfone or chelating or chelate or lactate)) and (510/17\$.ccls.)	USPAT; US-PGPUB; EPO; JPO; DERWENT; IBM_TDB	2003/01/15 13:06

-----claim tree-----

1----2----3----8

+-----13

+-----12

+-----11

+-----10

+-----9

+-----7

+-----5

+-----4

6

14----15----16----17

+-----22

+-----21

+-----20

+-----19

+-----18

23----24----25

+-----31

+-----30

+-----29

+-----28

+-----27

+-----26

-----112-----

-----best-----

6245155

5780406

5705089

5334332

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Re30796

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5614027

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6479374

6110451

6495573

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4549968

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6482567

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6479039
5744504
6491970
6482848
6506419
6498163
6506738
4350606
6458849
3582401
3160539

-----classlist-----

134/2
510/176
134/3
510/175
134/40
252/793
134/38
252/794
134/41
510/499
510/212
134/28
134/902
510/434
430/329
510/500
134/13
430/331
510/202
252/791
510/505
510/437
510/407
510/492
528/21
510/254
510/245
510/259
430/258
428/447
134/29
427/132
427/131
427/130
510/435
510/257
510/433
134/12
106/28712
510/423
430/330
528/14
528/34
427/387
134/34
510/493
134/2217
134/42
134/2219
510/504
525/446
510/258
134/2216
510/108
528/23
134/36
106/28714
427/4342
134/254
430/256

427/289
 427/420
 528/39
 510/272
 510/501
 106/28716
 510/489
 427/404
 525/342
 427/4071
 430/326
 510/255

-----keywords-----

2-amino-2-ethoxy sulfone solvent amount water amount of water deionized water percent water weight percent water water hydrofluoric hydrogen ammonium fluoride ammonium hydrogen fluoride ammonium fluoride hydrogen fluoride sulfoxide dmsol dimethylsulfoxide chelating co-solvent alkanolamine ethanolamine catechol gallic propylene carbonate pyrrolidone ethyl lactate acetic acid methyl acetate methyl lactate hydrogen fluoride ammonium hydrogen silicon oxide etching silicon substrate removing photoresist removing polyethylene glycol polyethylene N-methyl pyrrolidone pyrrolidone N-methyl propylene carbonate carbonate propylene glycol propylene ethyl lactate ethylene glycol ethyl acetate methyl lactate lactate methyl acetate acid methyl acetic acid acetic co-solvent hydroxide gallic acid chelating etching metal etching aqueous solution triethanolamine diethanolamine formate monoethanolamine ethanol ammonium fluoride alkyl organic fluoride compounds fluoride substrates etch residue residue etch cleaning stripping photoresist photoresist stripping claimed sulfoxide corresponding sub sulfone independently dimethylsulfoxide hydroxylamine hydrazine diethylhydroxylamine choline tetramethylammonium monoisopropanolamine ratio solvent/amine solution gallic catechol hydrogen tetrabutyl phosphonium silicon dicarbethoxyhydrazine coating compounds solvent amine ammonium aqueous metal oxide acid methyl acetate ethyl ethylene glycol diacetate methoxyethoxyethanol monolaurate appended claims organic amine ranges concentrations carboxylic ester groups ketone beneficial hydroxyl groups nitrogen carbonyl hydroxyl functional groups substituted chosen severe ingredients dissolution blend wetting balance fluoride salt benefits photoresist stripper rinse ambient immersed agitation stirring cleaning substrate derivatives hydroxides magnesium sodium potassium cleaning solution tartaric succinic oxalic malonic malic benzoic propionic formic organic acids hydrochloric acid hydrochloric phosphoric sulfuric silicone amphoteric nonionic anionic cationic cationic classified surfactant vinyl alcohol improves zinc nitrate zinc potassium nitrate polyols amides deionized listed N-alkyl stated ratios dimethyl sulfoxide dimethyl desirably inhibit corrosion effectiveness glycols butyl acetate acetate ethyl discloses liquid phase ammonia evaporation extent contacting

-----references-----

----- 6245155
 classes:1 134/3 1 134/12 1 134/2 1 134/2216 1 134/2217 1 134/2219 1 134/254 1 134/34 1 134/36 1 134/41 1 134/902 1 510/175 1 510/176 1 510/245 1 510/254 1 510/257 1 510/259 1 510/504
 score: 965

keywords: water;chelating;alkanolamine;catechol;substrate;removing photoresist;removing;ethylene glycol;hydroxide;chelating;etching;aqueous solution;alkyl;organic;fluoride;substrates;residue;etch;cleaning;photoresist;stripping;claimed;hydroxylamine;tetramethylammonium;solution;catechol;silicon;compounds;solvent;amine;ammonium;aqueous;metal;oxide;acid;methyl;ethylene;glycol;hydroxyl;balance;photoresist stripper;rinse;derivatives;hydroxides;succinic;nonionic;cationic;surfactant;nitrate;deionized;discloses;contacting;elevated;consists;amines;photoresist stripping;inhibit;teaches;water-soluble;solubility;anionic;fluorine;plasma ashing;spraying;published;quaternary ammonium;quaternary;ammonium hydroxide;ineffective;issued;Films;compositions used;rinsed;alkaline;aluminum alloys;frequently;acidic;tungsten;titanium;aluminum;involves;alkanolamines;oxygen plasma;oxygen;

United States Patent: 6,245,155
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FULL TEXT FORMAT-->

 (1 of 1)

United States Patent
 6,245,155

Leon

, et al.

June 12, 2001

Method for **removing photoresist** and plasma **etch **residue**s

Abstract

A method for the **removing** of plasma **etch **residue**s on a **substrate** comprising

the steps of: (i) contacting the **substrate** with a **cleaning** composition, and (ii) contacting the **substrate** with ozonated water. The preferred **cleaning** composition has a pH from 2 to 6 and comprises: (A) water; (B) at least one selected hydroxylammonium compound; and (C) at least one basic compound; and optionally (D) a **chelating** stabilizer; and optionally (E) a surfactant.

Inventors:

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Honda; Kenji (Barrington, RI);
Rothgery; Eugene F. (North Branford, CT)

Assignee:

Arch Specialty Chemicals, Inc. (Norwalk, CT)

Appl. No.:

055630

Filed:

April 6, 1998

Current U.S. Class:

1

- from about 2 to about 4.
- 3. The method of claim 1, wherein said **substrate** is contacted with said aqueous based **cleaning** composition by spraying said **substrate** with said **cleaning** composition.
- 4. The method of claim 1, wherein said **substrate** is rinsed with said ozonated water by spray rinsing.
- 5. The method of claim 1, further comprising a step of subjecting said **substrate** to dry **stripping**.
- 6. The method of claim 1, further comprising a step of subjecting said **substrate** to **organic** wet **stripping**.
- 7. The method of claim 1, wherein said acidic hydroxylammonium compound is selected from the group consisting of hydroxylammonium salts of the formula:
n(NR.sub.1 R.sub.2 R.sub.3 OH).sup.+ (X.sup.-n)
wherein R.sub.1, R.sub.2 and R.sub.3 are individually selected from hydrogen, lower **alkyl** groups having 1 to 4 carbon atoms, lower alkoxy groups having 1 to 4 carbon atoms, hydroxyl and hydroxyl-substituted lower **alkyl** groups having 1 to 4 carbon a
 - onium phosphate, hydroxylammonium oxalate, hydroxylammonium citrate, hydroxylammonium **fluoride** and hydroxylammonium chloride.
- 9. The method of claim 1, wherein the amount of hydroxylammonium compound is from about 1% to about 70% by weight of said **cleaning** composition.
- 10. The method of claim 1, wherein said amine is selected from the group consisting of hydroxylamine, **monoethanolamine**, **diethanolamine**, **triethanolamine**, diethyleneglycolamine, and N-hydroxyethylpiperazine.
- 11. The method of claim 1, wherein said quaternary ammonium **hydroxide** is selected from the group consisting of tetramethylammonium **hydroxide**, tetraethyl-ammonium **hydroxide**, trimethyl hydroxyethylammonium **hydroxide**, methyl tri (hydroxyethyl) ammonium **hydroxide**, tetra(hydroxyethyl)ammonium **hydroxide**, and benzyl trimethylammonium **hydroxide**.
- 12. The method of claim 1, wherein the amount of said basic compound in the **cleaning** composition is from about 0.01% to about 10% by w
 - actants and anionic surfactants.
- 15. The method of claim 1, wherein said **cleaning** composition further comprises a corrosion inhibitor selected from the group consisting of: sugar alcohols and catechol.
- 16. The method of claim 5, wherein said dry **stripping** is a plasma oxygen ashing.
- 17. The method of claim 5, wherein said step of dry **stripping** is conducted prior to contacting said **substrate** with said aqueous-based **cleaning** composition.

Description

BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention relates to a **cleaning** method for use in microelectronics manufacturing, and more particularly to a non-corrosive method for **removing photoresist** and plasma **etch residue**s formed on wafer **substrates** after plasma **etching** of metal layers or oxide layers deposited on the **substrates**.

2. Brief Description of Art

In the manufacture of microcircuits, positive **photoresist**s are used as an intermediate mask for transferring an original

----- 5780406
 classes:1 510/175 1 134/13 1 134/2 1 134/3 1 134/38 1 134/40 1 134/41 1 134/42 1 510/176 1 510/437
 score: 928

keywords: deionized water;water;chelating;alkanolamine;catechol;substrate;removing;ethylene glycol;hydroxide;chelating;etching;aqueous solution;alkyl;organic;substrates;etch residue;residue;etch;cleaning;photoresist;stripping;claimed;hydroxylamine;tetramethylammonium;ratio;solution;catechol;compounds;solvent;amine;ammonium;aqueous;metal;oxide;acid;methyl;ethylene;glycol;nitrogen;hydroxyl;blend;balance;photoresist stripper;rinse;immersed;derivatives;hydroxides;cleaning solution;nonionic;cationic;surfactant;nitrate;deionized;discloses;contacting;consists;amines;photoresist stripping;adding;teaches;water-soluble;anionic;plasma ashing;vapor;metal oxide;published;quaternary ammonium;quaternary;ammonium hydroxide;ineffective;Films;temperatures;alkaline;dissolving;acidic;tungsten;titanium;aluminum;involves;tetramethylammonium hydroxide;alkanolamines;oxygen plasma;oxygen;

- d quaternary ammonium **hydroxide** radical;
 and n is the valence of X and is from 1 to 3; and
- (C) at least one basic compound selected from the group consisting of amines and quaternary ammonium **hydroxide**s.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS OF THE INVENTION

It has been found that selective plasma **etching residue** removal with no **substrate** metal layer attack can be achieved by the combination of (1) adjusting a pH of the **cleaning** solution and (2) use of selected basic and acidic components. That is, the **cleaning** composition of the present invention comprises admixture of a basic component and an acidic component so as to set the pH of the solution in an acidic range of about 2 to about 6. The basic component used in this case is selected from amines and quaternary ammonium **hydroxide**s, while the acidic component is selected from hydroxylammonium salts which also contribute to enhance the removal of the plasma **etching** resi

- **ethanol**amine, **diethanolamine**, **triethanolamine**, diethyleneglycolamine, N-hydroxyethylpiperazine, and the like.

Quaternary ammonium **hydroxide**s useful in the composition of the invention include tetraalkylammonium **hydroxide**s having **alkyl** groups of methyl, ethyl, propyl, butyl, hydroxyethyl, benzyl, and the combinations thereof (e.g., such as tetramethylammonium **hydroxide** (hereinafter referred to as TMAH), tetraethylammonium **hydroxide**, trimethyl hydroxyethylammonium **hydroxide**, methyl tri(hydroxyethyl)ammonium **hydroxide**, tetra(hydroxyethyl)ammonium **hydroxide**, benzyl trimethylammonium **hydroxide**, and the like). Additionally, a combination of ammonium **hydroxide** with one or more quaternary ammonium **hydroxide**s may also be used.

Preferably, the basic compound is present in the composition of the invention in the range of about 0.01% to about 10% by weight, and the balance of the composition is water.

- A **chelating** stabilizer may be option
- ange of about 0.1 ppm to about 30 ppm by weight to a total weight of the **cleaning** composition of the present invention.

The **cleaning** composition of the present invention can be used either in combination with the oxygen plasma ashing or with a conventional wet **photoresist stripping** solution. The **cleaning** composition of this invention is not specifically designed to remove **photoresist** films from wafer **substrates**. Rather, the present **cleaning** composition is designed to remove plasma **etching residue**s after **removing photoresist**s by the dry or wet **stripping** methods. The **cleaning** composition of the invention can also be used to replace the **organic** solvent-based post-strip rinse because it is a non-corrosive and weakly acidic **aqueous solution** that will not harm the environment. Thus, the **cleaning** composition may be disposed of in a regular drain system for aqueous chemical wastes.

The following Examples further illustrate the present

----- 5705089
 classes:1 252/791 1 252/792 1 252/793 1 252/794 1 252/795
 score: 906

keywords: water;hydrofluoric;chelating;substrate;removing;polyethylene glycol;polyethylene;propylene;acetic;hydroxide;chelating;aqueous solution;alkyl;organic;substrates;cleaning;claimed;corresponding;choline;t

etramethylammonium; solution; hydrogen; silicon; ammonium; aqueous; metal; oxide; acid; ethylene; glycol; carboxylic; carbonyl; substituted; wetting; tartaric; oxalic; hydrochloric acid; hydrochloric; phosphoric; sulfuric; nonionic; cationic; surfactant; improves; glycols; ammonia; novel; substances; adding; fluorinated; water-soluble organic; water-soluble; citric; anionic; ammonium salt; quaternary ammonium; quaternary; rinsed; improved cleaning; problems; acidic; tetramethylammonium hydroxide;

- added additives shown in Table 5 in proportions shown in Table 5, respectively, at 85.degree. C. for 10 minutes for **cleaning**. Then, after rinsed with ultrapure water, the silicon **substrate** was examined by using a scanning electron microscope to check effect of **removing** silica particles on the silicon **substrate**. Results obtained are shown in Table 5.

Comparative Example 8

Similar procedures as in Examples 14 and 16 were repeated using lactic acid as an additive. Results obtained are shown in Table 5.

Comparative Example 9

Similar procedures as in Examples 14 and 16 were repeated using no additive. Result obtained are shown in Table 5.

TABLE5

Run No.	Additive	Number of Particles on Surface of **substrate** (number/substrate)		Removal		Ratio
		Amount (ppm)	**cleaning**	Before	After	

- and ultrapure water were mixed in proportions of 4:1:20 by weight, and additives shown in Table 6 were further added to the resulting mixture to prepare **cleaning** fluids. A silicon **substrate** of 3 inches in diameter of which native oxide was removed with diluted hydrofluoric acid treatment was dipped in the **cleaning** fluid thus obtained at 85.degree. C. for 10 minutes for **cleaning**. After rinsed with ultrapure water, this was examined for presence of residual additives based on absorptions of **organic** substances by using XPS analysis (X-ray Photo Spectroscopy) and ATR-IR analysis (Attenuated Total Reflection Infrared Spectroscopy). Results obtained are shown in Table 6.

Comparative Examples 10 and 11

Similar procedures as in Examples 17 and 19 were repeated using **polyethylene** glycol **alkyl** ether (surfactant, Comparative Example 10) or **alkyl**benzenesulfonic acid (surfactant, Comparative Example 11) as an additive. Results obtain

----- 5334332

classes:1 510/175 1 134/2 1 134/40 1 510/108 1 510/245 1 510/254 1 510/434 1 510/489 1 510/492 1 510/505
score: 849

keywords: deionized water; water; chelating; alkanolamine; silicon oxide; substrate; removing; propylene glycol; propylene; ethylene glycol; hydroxide; chelating; etching; alkyl; organic; substrates; etch residue; residue; etch; cleaning; photoresist; stripping; corresponding; hydroxylamine; hydrazine; tetramethylammonium; ratio; solution; silicon; coating; compounds; solvent; ammonium; metal; oxide; acid; ethyl; ethylene; glycol; carboxylic; severe; balance; rinse; ambient; immersed; agitation; derivatives; potassium; deionized; effectiveness; extent; contacting; amines; photoresist stripping; metal ions; oxide etching; integrated circuit; synergistic; contamination; plasma ashing; metal oxide; ammonium salt; ineffective; Films; polymers; bath; coatings; rinsed; temperatures; alkaline; chemically; polymeric; poor; tungsten; titanium; aluminum; involves; involving; tetramethylammonium hydroxide; alkanolamines; oxygen plasma; oxygen; species; reacted; interaction; undesirable;

- onamines. The alkanol group of the amines preferably has from 1 to 5 carbon atoms. Preferred alkanolamines suitable for use in the present invention can be represented by the chemical formula R.sub.1 R.sub.2 --N--CH.sub.2 CH.sub.2 --O--R.sub.3 wherein R.sub.1 and R.sub.2 can be H, CH.sub.3, CH.sub.3 CH.sub.2 or CH.sub.2 CH.sub.2 OH and R.sub.3 is CH.sub.2 CH.sub.2 OH. Examples of suitable alkanolamines include **monoethanolamine**, **diethanolamine**, **triethanolamine**, tertiarybutyldiethanolamine isopropanolamine, 2-amino-1-propanol, 3-amino-1-propanol, isobutanolamine, 2-amino-2-ethoxyethanol, and 2-amino-2-ethoxy-propanol. Polar solvents suitable for use in the **stripping** composition of the present invention include **ethylene glycol**, **ethylene glycol alkyl** ether, diethylene glycol **alkyl** ether, triethylene glycol **alkyl** ether, **propylene glycol**, **propylene glycol alkyl** ether, dipropylene glycol **alkyl** ether, tripropylene

- glycol **alkyl** ether, N-su
like salts would not
therefore be suitable for use based upon the understood mechanism of ionic
contamination in a microcircuit as caused by **cleaning** and set forth above.
As evident from the above formula, the carboxylic acid may be mono-, di-
or trisubstituted rather than tetra-substituted.
- A preferred **cleaning** composition of the present invention includes 30% by
weight hydroxylamine, 25% by weight 2-amino-2-ethoxyethanol, 5% by weight
1,2-dihydroxybenzene and 50% by weight of water.
- The **stripping** compositions of the present composition are effective in
removing a wide range of positive **photoresist**s but are particularly useful
in **removing photoresist**s commonly consisting of an ortho-naphthoquinone
diazide sulfonic acid ester or amide sensitizer with novolak-type binders
or resins. Examples of commercially available **photoresist** compositions
which the **stripping** compositions of the present invention effectively
remove from a su
- ubstrates themselves include metal **substrates** such as aluminum,
titanium/tungsten, aluminum/silicon, aluminum/silicon/copper; and
substrates such as **silicon oxide**, silicon nitride, and gallium/arsenide;
and plastic **substrates** such as polycarbonate.
- The **cleaning** compositions of the present invention are also effective in
removing organometallic and metal oxide **residue** generated on the **substrate**
of the **etching** equipment utilized. Examples of commercially available
etching equipment include Lam Research, Tegal, Electrotech, Applied
Material, Tokyo Electron, Hitachi and the like.
- The method of **removing** a resist or other material from a **substrate** using
the **stripping** compositions of the present invention involves contacting a
substrate having a resist thereon with a **stripping** composition of the
present invention for a time and at a temperature sufficient to remove the
resist. The time and temperature are determined based on the particula

----- 5466389
classes:1 510/175 1 134/2 1 134/42 1 510/108 1 510/421 1 510/423 1 510/435 1 510/499 1 510/506
score: 836

keywords: deionized water;water;chelating;alkanolamine;acetic acid;silicon oxide;substrate;removing;acetic
acid;acetic;hydroxide;chelating;etching;ethanol;alkyl;organic;substrates;etch residue;residue;etch;clea
ning;photoresist;claimed;hydrazine;choline;solution;hydrogen;tetrabutyl;silicon;coating;compounds;ammoniu
m;aqueous;metal;oxide;acid;acetate;glycol;beneficial;wetting;balance;benefits;hydroxides;cleaning solutio
n;tartaric;oxalic;malonic;organic acids;hydrochloric acid;hydrochloric;nonionic;surfactant;potassium;nitr
ate;deionized;listed;ammonia;contacting;amines;metal ions;inhibit;environmental;integrated circuit;silico
n dioxide;dioxide;fluorinated;citric;contamination;plasma ashing;vapor;quaternary ammonium;quaternary;eth
ers;phase;ammonium hydroxide;tetraalkyl ammonium;nitric acid;nitric;dimensions;formulation;bath;rinsed;fo
rmulations;alkaline;dissolving;protective;wafer substrate;underlying metal;titanium;aluminum;involves;alk
anolamines;undesirable;

Base	Surfactant	R.sub.z	
Concentration %	Concentration %	(Angstroms)	pH
tetraethylammonium	none	5,000	13.1
hydroxide (1.0)			
tetraethylammonium	0.1	3,300	13.2
hydroxide (1.0)			
tetraethylammonium	0.1	<25	9.2
hydroxide (1.0)			
choline (1.0)	none	4,300	12.9
choline (1.0)	0.1	250	13.1
choline (1.0)	0.1	<25	9.2
2-aminoethanol (2.0)	none	16,000	11.9
2-aminoethanol (2.0)	0.1	1,200	12.0

2-aminoethanol (2.0)
 0.1 <25 9.2
 guanidine **carbonate**
 none 6,400 11.5

(2.0)

guani

- anium and then
 titanium nitride;

b. coating with a **silicon oxide** dielectric using chemical vapor deposition;

c. lithographic patterning of 0.5 to 1.0 microns vias using a **photoresist** material;

d. pattern transfer to the dielectric layer using reactive ion **etching**;

e. plasma ashing to remove most of the residual **photoresist**.

The resulting patterned wafers were contaminated with a considerable amount of residual ash that comprised a mixture of **organic** materials from the **photoresist** and inorganic materials from the underlying metal layer that had been partially exposed during step (d). The use of the hereinbefore described cleaner bath formulation as well as the following controlled pH alkaline **cleaning** solutions, all of which contained water, tetramethyl ammonium **hydroxide**, a nonionic surfactant (either 3,5-dimethylhexyne-3-ol or 1-butoxy-2-propanol), and a **chelating** agent (EDTA) and one of the following listed pH reducing components, removed this residual ash without damaging any silicon or metal features that were present.

pH Reducing Component	pH
Ammonium acetate	9.3-10.0
Ammonium acetate, ammonium **hydroxide**	9-10
acetic acid, ammonium **hydroxide**	9.5-10
acetic acid, 1-amino-2-propanol	9.5-10
acetic acid, hydrogen peroxide	9.3
Ammonium acetate, hydrogen peroxide	9.5
Ammonium acetate, nitric acid	9.5
Ammonium nitrate	8.9-10.0
Ammonium chloride, ammonium acetate, hydrogen peroxide	9.6-10.0
Ammonium chloride, ammonium acetate, ammonium periodate	9-10
Ammonium chloride, ammonium acetate, ammonium nitrate	9.4-10.0

----- 5308745
 classes:1 430/329 1 430/256 1 430/260 1 430/311 1 430/325 1 430/326
 score: 808

keywords: deionized water;water;hydrofluoric;sulfoxide;dms;acetic acid;substrate;removing;polyethylene;carbonate;acetic acid;acetic;aqueous solution;alkyl;organic;substrates;stripping photoresist;photoresist;stripping;sulfoxide;hydroxylamine;monoisopropanolamine;solution;hydrogen;compounds;solvent;amine;ammonium;aqueous;metal;acid;methyl;ethyl;glycol;carboxylic;beneficial;substituted;photoresist stripper;rinse;derivatives;hydroxides;organic acids;amides;deionized;listed;dimethyl sulfoxide;dimethyl;extent;amines;photoresist stripping;environmental;integrated circuit;adding;solubility;ethers;issued;Films;formulation;formulations;alkaline;neutralized;titanium;aluminum;interaction;undesirable;

- d or substantially eliminate metal corrosion on microcircuit **substrates** containing metals and coated with cross-linked or hardened **photoresist** resin. This invention also relates to a process for **stripping** **photoresist** from cross-linked or hardened **substrates** employing alkaline-containing **photoresist stripping** compositions without incurring any substantial metal corrosion in microcircuit **substrates** containing metal and which provide greatly improved strip rates, with consequent decrease in strip time, compared to **stripping** compositions not containing the alkaline component.

BACKGROUND OF THE INVENTION

An integral part of microelectronic fabrication is the use of **photoresist**s to transfer an image from a mask or reticle to the desired circuit layer.

After the desired image transfer has been achieved, the **photoresist** is removed by **stripping** before proceeding to some subsequent process step. Since about 1980, amides and mixtures of

----- 4111767
classes:1 205/717
score: 798

keywords: acetic acid;removing;polyethylene;acetic acid;acetic;hydroxide;aqueous solution;monoethanolamine;organic;stripping;claimed;hydroxylamine;hydrazine;solution;coating;compounds;amine;ammonium;aqueous;metal;acid;ethyl;ethylene;immersed;tartaric;succinic;malic;organic acids;hydrochloric acid;hydrochloric;sulfuric;potassium;nitrate;substances;adding;water-soluble;citric acid;citric;nitric acid;nitric;bath;coating;s;chemically;dissolving;substance;titanium;species;

- rrophoretic coatings from stainless steel base materials almost without dissolving the base materials.
The object of this invention can be achieved by adding about 1 to about 100 g/l of at least one reducing compound selected from the group consisting of dimethylformamide, formaldehyde, thiosulfates, sulfites, hydrazine, hydroxylamine and dimethylaminoboron to a **stripping** composition consisting of a nitrate, a chlorine ion donor, and an amine compound and/or an **organic** acid having a buffering action, and adjusting the resulting bath to a pH of 6.0 to 9.5.
Stated more specifically, this invention provides an electrolytic **stripping** bath for **removing** metal coatings from stainless steel base materials comprising about 50 to about 500 g/l of a water-soluble nitrate, about 1 to about 200 g/l of a chlorine ion donor, about 0.5 to about 200 g/l of at least one of amine compounds and **organic** acids having a buffering action, and about 1
- lyl chloride and chloral hydrate. Of these examples, sodium chloride, ammonium chloride, chloral hydrate and ethylene chlorohydrin are preferable to use. The chlorine ion donor is used in an amount of about 1 to about 200 g/l, preferably about 5 to about 100 g/l.
Examples of amine compounds and **organic** acids having a buffering action are ammonia, ethylenediamine, diaminopropane, diethylenetriamine, triethylenetetramine, tetraethylenepentamine, **monoethanolamine**, **diethanolamine**, **triethanolamine**, cyclohexylamine, aniline, toluidine, dimethylaniline, sulfanilic acid, urea, glycine, aspartic acid, alanine, glutamic acid, aminobenzoic acid, aminosuccinic acid, iminodiacetic acid, nitrilotriacetic acid, quadrol, ethylenediaminetetraacetic acid and like amine compounds, and **acetic acid**, succinic acid, itaconic acid, maleic acid, fumaric acid, phthalic acid, citric acid, malic acid, gluconic acid, glycolic acid, lactic acid, tartaric acid

----- 6503633
classes:1 428/447 1 427/387 1 528/21 1 528/23 1 528/39 1 106/28712 1 106/28714 1 106/28716
score: 793

keywords: amount of water;water;hydrofluoric;gallic;substrate;polyethylene glycol;polyethylene;propylene glycol;propylene;ethylene glycol;acetic;hydroxide;etching;alkyl;organic;substrates;claimed;independently;tetramethylammonium;solution;gallic;hydrogen;silicon;coating;compounds;solvent;aqueous;oxide;acid;methyl;ethyl;ethylene;glycol;ketone;nitrogen;ingredients;derivatives;succinic;oxalic;malonic;malic;benzoic;propionic;formic;organic acids;hydrochloric;phosphoric;sulfuric;silicone;amphoteric;nonionic;cationic;surfactant;potassium;dimethyl;ammonia;satisfactory;adding;citric;fluorine;vapor;published;ethers;hydrophilic;nitrilic acid;nitric;dielectric constant;Films;polymers;isopropanol;aromatic;protective;frequently;problems;poor;aluminum;tetramethylammonium hydroxide;oxygen;reacted;

- ulator to a value within that range produces the effect that the composition obtained has improved storage stability.
The pH regulator is used in an amount suitably selected so that the pH of the composition becomes a value within that range.
Organic Solvent (B)
The composition for film formation of the invention comprises the product of hydrolysis and condensation (A) usually dissolved or dispersed in an **organic** solvent (B).
This **organic** solvent (B) may comprise at least one member selected from the group consisting of alcohol solvents, ketone solvents, amide solvents, ester solvents, and aprotic solvents.
Examples of the alcohol solvents include monohydric alcohols such as methanol, **ethanol**, n-propanol, isopropanol, n-butanol, isobutanol,

Examples of the ketone solvents include acetone, methyl ethyl ketone, methyl n-propyl ketone, methyl n-butyl ketone, diethyl ketone, methyl isobutyl ketone, methyl n-pentyl ketone, ethyl n-butyl ketone, methyl n-hexyl ketone, diisobutyl ketone, trimethylnonanone, cyclohexanone, 2-hexanone, methylcyclohexanone, 2,4-pentanedione, acetylacetone, acetophenone, and fenchone. Examples thereof further include .beta.-diketones such as acetylacetone, 2,4-hexanedione, 2,4-heptanedione, 3,5-heptanedione, 2

Examples of the ester solvents include diethyl **carbonate**, ethylene **carbonate**, **propylene carbonate**, **methyl acetate**, **ethyl acetate**, **gamma-butyrolactone**, **gamma-valerolactone**, **n-propyl acetate**, **isopropyl acetate**, **n-butyl acetate**, **isobutyl acetate**, **sec-butyl acetate**, **n-pentyl acetate**, **sec-pentyl acetate**, **3-methoxybutyl acetate**, **methylpentyl acetate**, **2-ethylbutyl acetate**, **2-ethylhexyl acetate**, **benzyl acetate**, **cyclohexyl acetate**, **methylcyclohexyl acetate**, **n-nonyl acetate**, **methyl acetoacetate**, **ethyl acetoacetate**, **ethylene glycol monomethyl ether acetate**, **ethylene glycol monoethyl ether acetate**, **diethylene glycol monomethyl ether acetate**, **diethylene glycol monoethyl ether acetate**, **thylpiperazine**, **N-methylimidazole**, **N-methyl-4-piperidone**, **N-methyl-2-piperidone**, **N-methyl-2-pyrrolidone**, **1,3-dimethyl-2-imidazolidinone**, and **1,3-dimethyltetrahydro-2(1H)-pyrimidinone**.

R.sub.10 O(CHCH.sub.3 CH.sub.2 O).sub.e R.sub.11 (5)
wherein R.sub.10 and R.sub.11 each independently represents a hydrogen atom
or a monovalent ****organic**** group selected from the group consisting of ****alkyl****
groups having 1 to 4 carbon atoms and CH.sub.3 CO--; and e is an integer
of 1 or 2.

In hydrolyzing and condensing the compounds (1) to (4) for preparing the composition for film formation of the invention, the same solvents as those enumerated above can be used.

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----- 5935920
classes:1 510/245 1 510/404 1 510/405 1 510/421 1 510/433 1 510/434 1 510/435 1 510/436
score: 788
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- e used as powders, as pastes rich in active substances or as more or less highly concentrated aqueous concentrates. 2 to 10% by weight ****aqueous solution**** are preferably used in the case of powders, 2 to 15% by weight ****aqueous solution**** are preferably used in the case of pastes and 5 to 15% by weight aqueous solutions are preferably used in the case of liquid formulations.

Page 10

surfactants can be combined with anionic surfactants. Accordingly, the present invention also encompasses processes in which, in addition to the formulations according to the invention, the aqueous **cleaning** solution additionally contains one or more anionic surfactants selected from at least one of the groups q) **alkyl** sulfates with a linear or branched C.sub.10-18 **alkyl** group, r) **alkyl** polyglycol ether sulfates with a linear or branched C.sub.10-18 **alkyl** group and 1 to 6 --CH.sub.2 --CH.sub.2 --

----- 4617251

classes:1 430/256 1 134/38 1 430/258 1 430/331 1 510/176 1 510/212 1 510/407 1 510/493 1 510/499 1 510/500 1 510/501 1 510/505
score: 761

keywords: deionized water;water;dms0;substrate;removing;ethanol;alkyl;organic;substrates;stripping;claimed;independently;tetramethylammonium;solution;silicon;coating;compounds;solvent;amine;aqueous;acid;acetate;glycol;ranges;ketone;ingredients;immersed;Stirring;deionized;N-alkyl;dimethyl;effectiveness;contacting;environmental;dioxide;bath;rinsed;temperatures;hydrocarbon;organic polymeric;polymeric;completion;problems;aluminum;involves;

- ganic polymeric material can be immersed in a **stripping** bath or the **stripping** composition can be sprayed over the surface of the **organic** polymeric material, as will be apparent to those skilled in the art.
- The **stripping** composition of the invention is effective in **removing** a wide variety of **organic** polymeric materials from **substrates**. Exemplificative **organic** polymeric materials include positive and negative resists, electron beam resists, x-ray resists, ion beam resists, as well as **organic** dielectric materials such as polyimide resins, and so forth. Specific examples of **organic** polymeric materials which can be removed in the practice of the invention include positive resists containing phenol formaldehyde resins or poly(p-vinylphenol); negative resists containing cyclized polyisoprene or poly(p-vinylphenol); and polymethylmethacrylate-containing resists which are useful as electron beam or deep UV resists. In particularly
- r each are summarized in Table I below. In each case, with the exception of Example 16, either all or virtually all of the resist was removed with the **stripping** composition utilized. In Example 16, there was a 50% removal; however, when this Example was repeated by employing an immersion time of about 10-15 minutes, 100% removal was achieved.
- In Table I, the following abbreviations are used:

For The Amine Compounds

AEE	2-(2-Aminoethoxy) ethanol
IBPA	3,3'-Iminobispropylamine
TEA	triethanolamine
MEA	monoethanolamine
DEA	diethanolamine
AEAE	2-(2-Aminoethylamino) ethanol
TT	Triethylenetetramine
APDEA	N--(3-Aminopropyl) diethanolamine

For the **organic** Polar Solvents

NMP	N--Methyl-2-Pyrr
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----- 6465368

classes:1 438/780 1 428/414 1 438/781 1 528/10 1 528/12
score: 753

keywords: amount of water;water;hydrofluoric;chelating;gallic;substrate;removing;polyethylene;propylene glycol;propylene;ethylene glycol;ethyl acetate;acetic;hydroxide;chelating;etching;aqueous solution;alkyl;organic;claimed;tetramethylammonium;solution;gallic;hydrogen;silicon;coating;compounds;solvent;ammonium;aqueous;metal;oxide;acid;methyl;acetate;ethyl;ethylene;glycol;ketone;nitrogen;Stirring;derivatives;tartaric;succinic;oxalic;malonic;malic;benzoic;propionic;formic;organic acids;hydrochloric;phosphoric;sulfuric;silicone;amphoteric;nonionic;cationic;potassium;dimethyl;butyl acetate;ammonia;evaporation;consists;amines;metal ions;adding;solubility;citric acid;citric;anionic;fluorine;vapor;hydrophilic;nitric acid;nitric;dielectric constant;Films;polymers;hydrocarbon;aromatic;alkaline;dissolving;acidic;poor;titanium;aluminum;tetramethylammonium hydroxide;oxygen;reacted;

- xtraction into water or an acidic ****aqueous solution****, followed by stirring, standing, and layer separation, is more preferable. When an acidic ****aqueous solution**** is used for the liquid-liquid extraction, it is desirable to repeat extraction several times using water to remove the acid.
- As required, a solvent for forming coating films may be added to the polymer component solution thus obtained. Then, the residual water and specific ****organic**** solvent is removed from the polymer component solution by vacuum distillation or the like.
- At least one solvent selected from the group consisting of alcohol solvents, ketone solvents, amide solvents, ester solvents, and non-protonic solvents can be used as the solvent for forming coating films.
- The following solvents can be given as examples of alcohol solvents: monohydric alcohols such as methanol, ****ethanol****, n-propanol, i-propanol, n-butanol, i-butanol, sec-butanol, t-butanol, n-pentanol, i-pen
- er, ****ethylene glycol**** monophenyl ether, ****ethylene glycol**** mono-2-ethylbutyl ether, diethylene glycol monomethyl ether, diethylene glycol monoethyl ether, diethylene glycol monopropyl ether, diethylene glycol monobutyl ether, diethylene glycol monohexyl ether, ****propylene glycol**** monomethyl ether, ****propylene glycol**** monoethyl ether, ****propylene glycol**** monopropyl ether, ****propylene glycol**** monobutyl ether, dipropylene glycol monomethyl ether, dipropylene glycol monoethyl ether, dipropylene glycol monopropyl ether, and the like.
- These alcohol solvents may be used either individually or in combination of two or more.
- Given as ketone solvents are acetone, methyl ethyl ketone, methyl n-propyl ketone, methyl n-butyl ketone, diethyl ketone, methyl i-butyl ketone, methyl n-pentyl ketone, ethyl n-butyl ketone, methyl n-hexyl ketone, di-i-butyl ketone, trimethylenonane, cyclohexanone, methyl cyclohexanone, 2,4-pentane dione, acetonyl acetone, acetophe
- N-ethylformamide, N,N-diethylformamide, acetamide, ****N-methyl****acetamide, N,N-dimethylacetamide, N-ethylacetamide, N,N-diethylacetamide, ****N-methyl****propionamide, ****N-methyl****pyrrolidone, N-formylmorpholine, N-formylpiperidine, N-formylpyrrolidine, N-acetylmorpholine, N-acetylpiperidine, N-acetylpyrrolidine, and the like can be given.
- These amide solvents may be used either individually or in combination of two or more.
- As ester solvents, diethyl ****carbonate****, ethylene ****carbonate****, ****propylene**** ****carbonate****, diethyl ****carbonate****, ****methyl acetate****, ****ethyl acetate****, .gamma.-butyrolactone, .gamma.-valerolactone, n-propyl acetate, i-propyl acetate, n-butyl acetate, i-butyl acetate, sec-butyl acetate, n-pentyl acetate, sec-pentyl acetate, 3-methoxybutyl acetate, methylpentyl acetate, 2-ethylbutyl acetate, 2-ethylhexyl acetate, benzyl acetate, cyclohexyl acetate, methylcyclohexyl acetate, n-nonyl acetate, methyl acetoacetate, ethyl acetoacetate, ethylene
- xide structure, polypropylene oxide structure, polytetramethylene oxide structure, polybutylene oxide structure, and the like can be given.
- Specific examples of the compound having a polyalkylene oxide structure include ether compounds such as polyoxymethylene ****alkyl**** ether, polyoxyethylene ****alkyl**** ether, polyoxyethylene ****alkyl****phenyl ether, polyoxyethylene sterol ether, polyoxyethylene lanolin derivatives, ethylene oxide derivatives of ****alkyl****phenol formalin condensate, polyoxyethylene polyoxypropylene block copolymers, and polyoxyethylene polyoxypropylene ****alkyl**** ethers; ether-ester compounds such as polyoxyethylene glyceride, polyoxyethylene sorbitan fatty acid ester, polyoxyethylene sorbitol fatty acid ester, and polyoxyethylene fatty acid alkanolamide sulfate; and ester compounds such as ****polyethylene**** glycol fatty acid ester, ****ethylene glycol**** fatty acid ester, fatty acid monoglyceride, polyglycerol fatty acid ester, sorbitan fat

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classes:1 428/447 1 525/342 1 525/446 1 528/14 1 528/21 1 528/34
score: 746

keywords: amount of water;water;hydrofluoric;gallic;acetic acid;substrate;polyethylene glycol;polyethylen
e;propylene glycol;propylene;ethylene glycol;acetic acid;acetic;hydroxide;alkyl;organic;substrates;claime
d;independently;tetramethylammonium;solution;gallic;hydrogen;silicon;coating;compounds;solvent;ammonium;a
queous;metal;oxide;acid;methyl;ethyl;ethylene;glycol;ketone;nitrogen;derivatives;hydroxides;succinic;oxal
ic;malonic;malic;benzoic;propionic;formic;organic acids;hydrochloric;phosphoric;sulfuric;silicone;amphote

ric;nonionic;cationic;surfactant;potassium;dimethyl;ammonia;amines;satisfactory;adding;fluorine;vapor;published;ethers;hydrophilic;nitric acid;nitric;dielectric constant;films;polymers;isopropanol;aromatic;protective;frequently;problems;poor;aluminum;tetramethylammonium hydroxide;oxygen;reacted;

- combination of two or more thereof.

Examples of the pH regulator include inorganic acids and **organic** acids.

Examples of the inorganic acids include hydrochloric acid, nitric acid, sulfuric acid, hydrofluoric acid, phosphoric acid, boric acid, and oxalic acid.

Examples of the **organic** acids include **acetic acid**, propionic acid, butanoic acid, pentanoic acid, hexanoic acid, heptanoic acid, octanoic acid, nonanoic acid, decanoic acid, oxalic acid, maleic acid, methylmalonic acid, adipic acid, sebacic acid, **gallic acid**, butyric acid, mellitic acid, arachidonic acid, shikimic acid, 2-ethylhexanoic acid, oleic acid, stearic acid, linoleic acid, linolenic acid, salicylic acid, benzoic acid, p-aminobenzoic acid, p-toluenesulfonic acid, benzenesulfonic acid, monochloroacetic acid, dichloroacetic acid, trichloroacetic acid, trifluoroacetic acid, formic acid, malonic acid, sulfonic acids, phthalic acid, fumaric acid, citric acid, tartaric acid

- 1 monoethyl ether, diethylene glycol monopropyl ether, diethylene glycol monobutyl ether, diethylene glycol monohexyl ether, **propylene glycol** monomethyl ether, **propylene glycol** monoethyl ether, **propylene glycol** monopropyl ether, **propylene glycol** monobutyl ether, dipropylene glycol monomethyl ether, dipropylene glycol monoethyl ether, and dipropylene glycol monopropyl ether.

Those alcohol solvents may be used alone or in combination of two or more thereof.

Examples of the ketone solvents include acetone, methyl ethyl ketone, methyl n-propyl ketone, methyl n-butyl ketone, diethyl ketone, methyl isobutyl ketone, methyl n-pentyl ketone, ethyl n-butyl ketone, methyl n-hexyl ketone, diisobutyl ketone, trimethylnonanone, cyclohexanone, 2-hexanone, methylcyclohexanone, 2,4-pentanedione, acetonylacetone, acetophenone, and fenchone. Examples thereof further include .beta.-diketones such as acetylacetone, 2,4-hexanedione, 2,4-heptan-

- ide, N-ethylacetamide, N,N-diethylacetamide, **N-methyl**propionamide, **N-methyl**pyrrolidone, N-formylmorpholine, N-formylpiperidine, N-formylpyrrolidine, N-acetylmorpholine, N-acetylpyrrolidine, and N-acetylpyrrolidine.

Those amide solvents may be used alone or in combination of two or more thereof.

Examples of the ester solvents include diethyl **carbonate**, ethylene **carbonate**, **propylene carbonate**, **methyl acetate**, **ethyl acetate**, .gamma.-butyrolactone, .gamma.-valerolactone, n-propyl acetate, isopropyl acetate, n-butyl acetate, isobutyl acetate, sec-butyl acetate, n-pentyl acetate, sec-pentyl acetate, 3-methoxybutyl acetate, methylpentyl acetate, 2-ethylbutyl acetate, 2-ethylhexyl acetate, benzyl acetate, cyclohexyl acetate, methylcyclohexyl acetate, n-nonyl acetate, methyl acetoacetate, ethyl acetoacetate, **ethylene glycol** monomethyl ether acetate, ethylene glycol monoethyl ether acetate, diethylene glycol monomethyl ether acetate

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classes:1 510/176 1 134/38 1 252/364 1 510/202 1 510/206 1 510/212 1 510/255 1 510/265 1 510/500
score: 738

keywords: water;substrate;removing;polyethylene;N-methyl;propylene;ethylene glycol;etching;diethanolamine;monoethanolamine;alkyl;organic;fluoride;substrates;etch;photoresist;stripping;claimed;solution;hydrogen;silicon;coating;compounds;solvent;amine;aqueous;metal;oxide;ethylene;glycol;carboxylic;nitrogen;hydroxyl;severe;ambient;nonionic;surfactant;amides;dimethyl;effectiveness;glycols;discloses;evaporation;contacting;amines;substances;environmental;silicon dioxide;dioxide;synergistic;fluorine;vapor;ethers;ineffective;formulation;coatings;temperatures;aromatic;chemically;dissolving;substance;polymeric;frequently;titanium;aluminum;

- h is incorporated herein by reference.

The **stripping** compositions of this invention are especially useful and advantageous for numerous reasons among which may be mentioned the following. The **stripping** compositions are water miscible, non-corrosive, non-flammable and of low toxicity to humans and the environment. Because

of the low ambient vapor pressure of the compositions they evidence substantially less evaporation than prior compositions and are non-reactive and environmentally compatible. The ****stripping**** compositions may be recycled for multiple use or easily disposed of in an environmentally safe manner without the necessity for burdensome safety precautions. Likewise, a portion of the stripped coatings may be readily removed as solids and collected for easy disposal. The ****stripping**** compositions of this invention evidence higher ****stripping**** efficiency at lower temperatures for a wide variety of coatings and ****substrates****.

- orrosion prevention on two different metal ****substrates****:

- 1) Pure 100% copper foil;
- 2) an Al/Cu (2%) metal alloy sputter deposited on top of a silicon ****substrate****.

The standard corrosion solvent used was of the following formulation:

Dimethylacetamide (DMA)	
	94% (w/w)
monoethanolamine (MEA)	6% (w/w)
Surfactant	0.2% added.

Previous tests with the following solvents were performed to assess corrosion propensity on pure copper ****substrates****. Attack was assessed by a change in color of the solvent to blue or blue-green indicating formation of a stable complex of oxidized (i.e. corroded) copper. The solvents included:

- 1) DMA
- 2) DMF
- 3) Tetrahydrofurfuryl alcohol (THFA)
- 4) ****propylene carbonate****
- 5) ****N-methylpyrrolidone (NMP)****

None of the solvent is showed any effect on the Cu at all.

Several amines (i.e., which are known to form stable complex

----- 6495506
 classes:1 510/417 1 510/365 1 510/422 1 510/505 1 510/506 1 424/401 1 424/402
 score: 724

keywords: amount of water;water;ethyl lactate;removing;polyethylene glycol;polyethylene;propylene glycol;propylene;ethyl lactate;ethylene glycol;lactate;alkyl;organic;residue;cleaning;independently;ratio;soluti on;hydrogen;compounds;solvent;amine;ammonium;aqueous;metal;oxide;acid;methyl;ethyl;ethylene;glycol;append ed claims;organic amine;ranges;concentrations;carboxylic;ingredients;balance;benefits;magnesium;tartaric; phosphoric;sulfuric;amphoteric;nonionic;surfactant;N-alkyl;desirably;discloses;amines;novel;substances;en vironmental;satisfactory;teaches;water-soluble;solubility;citric acid;citric;anionic;European;published;e thers;phase;cleaning formulation;formulation;bath;temperatures;formulations;hydrocarbon;aromatic;alkaline ;substance;polymeric;improved cleaning;acidic;aluminum;involves;reacted;undesirable;

- r, mono, di, tributylene glycol monobutyl ether, mono, di, tributylene glycol monopentyl ether and mono, di, tributylene glycol monohexyl ether, ****ethylene glycol**** monoacetate and dipropylene glycol propionate. When these glycol type cosurfactants are at a concentration of about 0.5 to about 14 weight %, more preferably about 2.0 weight % to about 10 weight % in combination with a water insoluble ****organic**** ester or non water soluble material such as terpene, essential oils which is at a concentration of at least 0.5 weight %, more preferably 1.5 weight % to about 8 wt. % one can form a microemulsion composition.

While all of the aforementioned glycol ether compounds provide the described stability, the most preferred cosurfactant compounds of each type, is diethylene glycol monobutyl ether. Other suitable water soluble cosurfactants are water soluble esters such as ****ethyl lactate**** and water soluble carbohydrates such as butyl gl

- roduct performance.

One such ingredient is an inorganic or ****organic**** salt of oxide of a multivalent metal cation, particularly Mg.sup.++. The metal salt or oxide provides several benefits including improved ****cleaning**** performance in dilute usage, particularly in soft water areas, and minimized amounts of perfume required to obtain the microemulsion state.

Magnesium sulfate, either anhydrous or hydrated (e.g., heptahydrate), is especially preferred as the magnesium salt. Good results also have been obtained with magnesium oxide, magnesium chloride, magnesium acetate, magnesium propionate and magnesium ****hydroxide****. These magnesium salts can be

used with formulations at neutral or acidic pH since magnesium **hydroxide** will not precipitate at these pH levels.

Although magnesium is the preferred multivalent metal from which the salts (inclusive of the oxide and **hydroxide**) are formed, other polyvalent metal ions also can be used provided that th

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classes:1 134/2 1 134/19 1 148/243 1 252/180 1 252/390 1 252/394 1 422/16 1 510/245 1 510/247 1 510/253
score: 716

keywords: water;gallic;removing;carbonate;hydroxide;gallic acid;aqueous solution;organic;cleaning;claimed;corresponding;independently;solution;gallic;amine;ammonium;aqueous;metal;oxide;acid;methyl;ranges;concentrations;substituted;rinse;agitation;derivatives;magnesium;organic acids;effectiveness;ammonia;contacting;consists;amines;resistant;adding;solubility;citric;vapor;ammonium salt;ammonium hydroxide;rinsed;alkaline;acidic;neutralized;

- ich water is evaporated or heat transfer occurs, insoluble salts deposit on the surfaces to form scale. Usually the deposits consist of calcium and magnesium salts and result from the use of hard water, although sometimes the scale is based on elements other than calcium or magnesium.

Common types of scale which deposit on metal surfaces include calcium sulfate (gypsum), calcium **carbonate** (limestone), complex calcium phosphate (hydroxy apatite) and calcium oxalate. In addition, scales containing a high iron content are also encountered.

It has previously been known to use inorganic or **organic** acids to remove scale deposits from ferrous metal heat exchange equipment. After descaling, the equipment is normally drained of the acidic solvent, flushed with water and residual acid, neutralized with an alkaline solution, drained again and finally flushed with water.

Alkali metal salts of **alkyl**enepolyamine polyacetic acids (hereafter APAPA), prototype ethylenediamine tetraacetic acid (EDTA), have also been used to remove scale from ferrous metal surfaces. They are ineffective, however, for **removing** iron oxide deposits at a pH above 8.

When aqueous acids or **aqueous solution**s of alkali metal salts of APAPA having a pH below 8 are used, the resulting cleaned ferrous metal surfaces are prone to oxidation or rusting, and the build-up of scale is again under way.

In accordance with the process of the present invention, scale and particularly iron oxide containing scale is removed from metal surfaces by a contacting treatment with an aqueous, alkaline, saline solution. As an essential feature, the solution employed contains at least one ammonium, amine or hydroxy **alkyl**amine salt of nitrilotriacetic acid (NTA), N-2-hydroxyethylamino diacetic acid (OHEtIDA), an **alkyl**enepolyamine polycarboxylic acid or mixture thereof. The contacting treatment is preferably carried

- lamino-, methyl ethylamino-, propylamino-, isopropylamino-, trimethylamino-, the isomeric butylamino-, the isomeric amylamino- the corresponding mono(alkanol)-, di(alkanol)-, tri(alkanol)amino- particularly the mono(ethanol)amino-, di(ethanol)amino- and tri(ethanol)amino- salts.

Representative of the ammoniated or aminated chelants set forth broadly above are those of ethylenediamine tetraacetic acid, diethylenetriamine pentaacetic acid, N-2-hydroxyethyl ethylenediamine triacetic acid, **propylene**-1,2-diamine tetraacetic acid, **propylene**-1,3-diamine tetraacetic acid, the isomeric butylenediamine tetraacetic acids, etc. When the chelants set forth above are ammoniated or aminated to a pH of 8 to 11, an average of one of the --CH.sub.2 CO.sub.2 H groups remains in the acid form since the bases used are not basic enough to form a fully substituted ammonium or amino salt of such chelants. In the formula given for such chelants,

----- 5279771

classes:1 510/212 1 134/38 1 430/331 1 510/176 1 510/202 1 510/407 1 510/499
score: 714

keywords: deionized water;water;alkanolamine;silicon oxide;substrate;removing;propylene glycol;propylene;ethylene glycol;etching;alkyl;substrates;residue;photoresist;stripping;hydroxylamine;hydrazine;silicon;coating;solvent;metal;oxide;acid;ethyl;ethylene;glycol;severe;rinse;immersed;agitation;derivatives;deionized;effectiveness;contacting;amines;integrated circuits;integrated circuit;synergistic;ineffective;Films;po

lymers;bath;coatings;rinsed;temperatures;polymeric;involves;involving;alkanolamines;oxygen;

- hydroxylamine. Further, the **stripping** composition can optionally contain one or more polar solvents. The addition of a polar solvent strengthens the effectiveness of the **stripping** composition. The **stripping** compositions allow for the clean removal of a resist from a **substrate**.
- The **stripping** compositions of the present invention are particularly suitable for **removing** a **photoresist** material from a **substrate**, in particular, during the manufacture of a semiconductor integrated circuit without adversely affecting or hindering subsequent manufacturing operation or process steps.
- Further, the **stripping** compositions of the present invention are suitable for **removing** cured polymer resists from a **substrate**, such as a partially or fully cured polyimide coating, and for **removing** organometallic polymers formed on a **substrate** during plasma **etching** processes.
- The method of **removing** a resist from a **substrate** using the **stripping** compositions o
- ub.1 and R.sub.2 can be H, CH.sub.3, CH.sub.3
- CH.sub.2 or CH.sub.2 CH.sub.2 OH and R.sub.3 is CH.sub.2 CH.sub.2 OH.
- Examples of suitable alkanolamines include **monoethanolamine**, **diethanolamine**, **triethanolamine**, tertiarybutyldiethanolamine isopropanolamine, 2-amino-1-propanol, 3-amino-1-propanol, isobutanolamine, 2-amino-(2-ethoxyethanol), and 2-amino(2-ethoxy)propanol.
- Polar solvents suitable for use in the present invention include ethylene glycol, **ethylene glycol alkyl** ether, diethylene glycol **alkyl** ether, triethylene glycol **alkyl** ether, **propylene** glycol, **propylene glycol alkyl** ether, dipropylene glycol **alkyl** ether, tripropylene glycol **alkyl** ether, N-substituted **pyrrolidone**, ethylenediamine, and ethylenetriamine. Additional polar solvents as known in the art can also be used in the composition of the present invention.
- The **stripping** compositions of the present composition are effective in **removing** a wide range of positive photoresi

----- 4239661
 classes:1 438/471 1 134/2 1 134/38 1 134/42 1 430/326 1 430/331 1 438/329 1 438/745 1 438/754 1 510/175 1 510/372 1 510/373 1 510/421 1 510/434 1 510/504
 score: 681

keywords: deionized water;water;hydrofluoric;silicon oxide;substrate;removing;carbonate;acetic;hydroxide; etching;aqueous solution;triethanolamine;alkyl;organic;etch;cleaning;photoresist;choline;ratio;solution;hydrogen;silicon;coating;compounds;solvent;ammonium;aqueous;metal;oxide;acid;glycol;carboxylic;nitrogen;immersed;hydrochloric acid;hydrochloric;phosphoric;sulfuric;nonionic;surfactant;potassium;deionized;ammonia;evaporation;extent;satisfactory;integrated circuit;adding;water-soluble;solubility;fluorine;phase;ammonium hydroxide;nitric acid;nitric;Films;waste;alkaline;dissolving;aluminum;oxygen;reacted;

- crystal plane of
- <100>. This example gave the same results as in Example 6. Moreover, the N type silicon semiconductor **substrate** itself was little etched by the above-mentioned cholin solution containing H.sub.2 O.sub.2 and NH.sub.4 CN.

EXAMPLE 13

A **silicon oxide** film was formed on the surface of an N type silicon semiconductor **substrate**. A positive-working **photoresist** film was coated on the **silicon oxide** film, using a solution prepared by dissolving the raw material of a positive-working **photoresist** in an **organic** solvent. The **photoresist** film was heated about 20 minutes at 80.degree. C. to evaporate the **organic** solvent and to suppress fogging. Later, a light was projected through a prescribed photomask aligned with the **photoresist**. Those portions of the **photoresist** on which a light was projected were etched by a 2.44% aqueous choline solution while those portions of the **photoresist** on which a light was not projected

----- 5750482
 classes:1 510/182 1 510/427 1 510/428 1 510/429 1 510/505 1 510/506
 score: 672

keywords: deionized water;water;chelating;acetic acid;carbonate;propylene glycol;propylene;ethylene glycol;acetic acid;acetic;hydroxide;chelating;monoethanolamine;alkyl;organic;cleaning;claimed;solvent;amine;ammonium;aqueous;metal;oxide;acid;methyl;acetate;ethyl;ethylene;glycol;concentrations;severe;ingredients;weighting;benefits;Stirring;succinic;propionic;organic acids;hydrochloric;phosphoric;sulfuric;amphoteric;noni

onic;surfactant;potassium;amides;deionized;dimethyl;effectiveness;glycols;discloses;ammonia;elevated;cons
ists;environmental;water-soluble;citric;anionic;European;quaternary ammonium;quaternary;phase;ammonium hy
droxide;isopropanol;temperatures;hydrocarbon;aromatic;alkaline;acidic;aluminum;

```

-          0.1176
Fluorad FC 129      0.05
Ammonia (29.4% active)
                  1.0
Potassium iodide    0.003
Water               <<< Q.S. 100% >>>

```

The Composition Q was tested as in Example 1 (but with 20 judges) against the Procter and Gamble product CINCH, which contains 3% **propylene glycol** monobutyl, ether and 7% isopropyl alcohol. CINCH also contained an alkalizing agent which is **monoethanolamine** and a surfactant. The test results indicated that Composition Q was superior to CINCH, notwithstanding the very high solvent contained in that commercial product (66 wins, 14 losses, 1.03 average **cleaning** score).

EXAMPLE 10

The compositions in the table which follows are illustrative of the present invention.

Ingredient	CONCENTRATION (Wt. %)									
	R	S	T	U	V	W	X	Y	Z	
	--	0.1								
				--		--	--	0.15		
Sodium dodecyl benzene sulfonate									--	
		0.1								
		0.1		--	0.08					
					0.2					
						0.15				
							0.25			
								--	0.2	
Nonoxynol-5	--									
		0.05								
		--	0.05							
				--	--	--	--	0.10		
									0.1	
Polyacrylic acid										
	--									
		0.02	--			--	0.02			
								--	0.05	
(MW = 3000)										
Fluorosurfactant										
	--									
		0.01								
		--	0.01							
				--	0.01					
						--	0.02			
								--		
acetic acid	--									
	--	0.15								

```

----- 6339054
classes:1 510/241 1 510/495 1 510/504
score: 647

```

keywords: water;hydrofluoric;chelating;gallic;acetic acid;substrate;removing;carbonate;propylene glycol;p
ropylene;ethylene glycol;acetic acid;acetic;hydroxide;gallic acid;chelating;aqueous solution;ethanol;alky
l;organic;substrates;residue;cleaning;independently;ratio;solution;gallic;compounds;solvent;amine;ammoniu
m;aqueous;metal;oxide;acid;methyl;ethylene;glycol;ranges;concentrations;carboxylic;beneficial;substituted
;chosen;ingredients;wetting;balance;benefits;rinse;hydroxides;cleaning solution;tartaric;succinic;oxalic;
malic;formic;organic acids;phosphoric;amphoteric;nonionic;cationic;surfactant;improves;potassium;listed;s
tated;dimethyl;desirably;extent;amines;metal ions;novel;satisfactory;recognized;water-soluble;citric acid
;citric;anionic;spraying;cleaning metal;carboxylate;quaternary ammonium;quaternary;phase;Films;formatio
n;waste;rinsed;temperatures;formulations;aromatic;alkaline;been removed;acidic;aluminum;species;reacted;u
ndesirable;

- trates, leaving water droplets. The
 the **substrates** look .about.95% dry in 2.25 mins. They feel dry to touch.
 20 PPM Half of the window fogs. No visible change on the
 substrate.
 100 PPM The window fogs from the top down and has droplet and water streams. Stable foam of 1.5".
 200 PPM Foam droplets or bubbles remain on the surface of the
 substrates. Foam level is 3". Water sheets rapidly. It took 1.5 mins drying time to reach the .about.95% dry surface.
 300 PPM 5-6 inches of foam. Signs of film on the **substrates**.
 500 PPM 6 inches of foam. Test was aborted at this point. While
 cleaning the machine a yellowish grease scum was detected on the back wall of the machine.

USE SOLUTION pH STUDY

pH of

Vo

- aqueous)
 3.50 Oxalic Acid dihydrate 99%
 6.00 Videt RFG Non-ionic Surfactant
 6.00 Linear Dodecyl Benzene Sulfonic Acid 97%
 2.50 Nonylphenol Ethoxylate 9.5 Mole
 7.50 Dehypon .TM. LS-54 non-ionic surfactant
 1.25 GlenSurf .TM. 42 cationic surfactant
 6.00 **propylene glycol** Monomethyl Ether
 2.00 Aliphatic Hydrocarbon
 100% TOTAL

This composition has found particular utility for touchless and friction-type car and truck washes, wheel **cleaning** and brightening processes in car washes. Carbon soil film removal in truck washing, rust stain removal in truck washing.

The function of the oxalic acid (as an example of the **organic** acid) and the phosphoric acid (as an example of the inorganic acid) have been noted as follows:

- 1) Phosphoric acid alone--No aluminum brightening
- 2) Oxalic acid alone--Some

----- 5614027

classes:1 134/2 1 134/29 1 134/40 1 510/255 1 510/258 1 510/259 1 510/272 1 510/423 1 510/433 1 510/434 1
 510/435 1 510/437 1 510/492 1 510/500 1 510/505
 score: 640

keywords: deionized water;water;alkanolamine;ethanolamine;pyrrolidone;substrate;removing;pyrrolidone;carbo
 onate;propylene;hydroxide;aqueous solution;alkyl;organic;substrates;cleaning;claimed;corresponding;ratio;
 solution;hydrogen;compounds;solvent;amine;ammonium;aqueous;metal;oxide;acid;ethylene;concentrations;carbo
 xylic;nitrogen;ingredients;balance;immersed;agitation;derivatives;magnesium;cleaning solution;nonionic;cl
 assified;surfactant;zinc;potassium;amides;deionized;N-alkyl;ratios;effectiveness;discloses;contacting;ele
 vated;consists;amines;metal ions;inhibit;novel;environmental;recognized;water-soluble;contamination;anion
 ic;cleaning metal;ethers;ketones;polymers;bath;waste;toxic;rinsed;formulations;hydrocarbon;alkaline;probl
 ems;aluminum;involves;species;undesirable;

- osphate esters of alkoxyated
 alcohols and sodium, potassium and ammonium salts of the **alkyl**
 sarcosinates. The hydrotropes are useful in maintaining the **organic**
 materials including the surfactant readily dispersed in the aqueous
 cleaning solution and, in particular, in an aqueous concentrate which is
 an especially preferred form of packaging the compositions of the
 invention and allow the user of the compositions to accurately provide the
 desired amount of **cleaning** composition into the aqueous wash solution. A
 particularly preferred hydrotrope is one that does not foam. Among the
 most useful of such hydrotropes are those which comprise the alkali metal
 salts of intermediate chain length linear **alkyl** monocarboxylic fatty
 acids, i.e., C.sub.7 -C.sub.13. Particularly preferred are the alkali
 metal octanoates and nonanoates.

The metal **cleaning** compositions of this invention comprise from about 20 to
 80 weight percent based on

- ution. It is to be understood that higher levels of the magnesium
 ion can be included in the **aqueous solution** but for the most part, higher
 levels than that described are not believed to add significantly to the
 anticorrosive effect. The zinc ion is effective at concentrations between

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1 and 1,000 ppm with respect to the aqueous ****cleaning**** solution. Again, higher levels of the zinc ion can be used but such higher levels are not believed to increase the anticorrosive effect. When used alone, the zinc ion is preferably utilized in concentrations of 25 to 100 ppm in the ****cleaning**** solution. When used with magnesium, the zinc ion levels are preferably from about 2 to 25 ppm based on the ****cleaning**** solution.

The aqueous metal ****cleaning**** solutions of the present invention are useful in ****removing**** a variety of contaminants from metal ****substrates**** as previously described. A useful method of ****cleaning**** such metal parts is in a parts washer. In parts

```
----- 4786578
classes:1 430/256 1 134/38 1 252/8 1 430/326 1 430/329 1 430/331
score: 632
```

keywords: water;substrate;removing photoresist;removing;etching;aqueous solution;triethanolamine;diethanolamine;alkyl;organic;substrates;residue;photoresist;stripping;claimed;ratio;solution;silicon;coating;solvent;aqueous;oxide;acid;ethylene;beneficial;dissolution;rinse;immersed;nonionic;surfactant;deionized;extend;amines;photoresist stripping;integrated circuits;water-soluble;solubility;contamination;bath;coatings;temperatures;

United States Patent: 4,786,578
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FULL TEXT FORMAT-->

(1of1)
United States Patent
4,786,578

Neisius

, et al.

November 22, 1988

Agent and method for the removal of **photoresist** and stripper **residue** from semiconductor **substrates**

Abstract

Photoresist and stripper ****residue****s can be rapidly and completely removed from semiconductor ****substrates**** after the ****stripping**** process with an aqueous post-rinsing agent which contains a nonionogenic surfactant and an ****organic**** base.

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Litters; Alois (Lampertheim Hofheim, DE)

Assignee:

Merck Patent Gesellschaft mit beschränkter Haftung (Darmstadt, DE)

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077970

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July 20, 1987

Foreign Application Priority Data

Jan 19, 1985[DE]3501675

Current U.S. Class:
430/256; 134/38; 252/8; 430/326; 430/329; 430/331
Intern'l Class:
G03C 011/12
Field of Search:

430/326,329,3

```
-      ein the **organic** base is **ethanol**amine, **diethanolamine** or
**triethanolamine**.
```

6. A method of claim 5, wherein the ****photoresist residue**** is a positive ****photoresist****.

7. A method of completely **removing photoresist** structures and **residue**s from a **substrate** which has been subjected to **photoresist** imaging, comprising treating the **substrate** with a **photoresist** stripper, **removing** the **substrate** from the stripper, and treating the **substrate** with a post-rinsing agent comprising effective amounts of a nonionic surfactant and an **organic** base in **aqueous solution**, said **organic** base being water soluble to a degree whereby it will form a water soluble salt with **alkyl benzenesulfonic acids** when the agent is contacted therewith.
8. A combination of (a) a **substrate** which has been subjected to **photoresist** **stripping** with a **photoresist stripping** agent and removed from said **photoresist stripping** agent and has resultant **photoresist** or stripper **residue** thereon, a

----- 4067690
 classes:1 422/16 1 252/387 1 252/392
 score: 629

keywords: water;N-methyl;carbonate;hydroxide;alkyl;claimed;hydroxylamine;hydrazine;ratio;solution;compounds;aqueous;metal;acid;methyl;severe;derivatives;hydrochloric;sulfuric;effectiveness;ammonia;elevated;con-
 ist;s;novel;dioxide;adding;water-soluble;nitric acid;nitric;toxic;temperatures;alkaline;acidic;involves;ox-
 ygen;

- EHA) was determined at 68.degree. F. without a catalyst and with a catalyst copper **carbonate** in a ratio of DEHA to catalyst of 100:1 parts by weight. A dosage of 10% excess DEHA was used based on 1 ppm of DEHA is required per 1 ppm of dissolved oxygen. The pH of the testing solution was adjusted by using dilute sodium **hydroxide** solution. The following results were recorded:

TABLE 6

N,N-DIETHYLHYDROXYLAMINE AND OXYGEN REACTION RATE AT 68.degree. F. AND pH 11 Time in DISSOLVED OXYGEN, mg/liter		
Minutes	No Catalyst	Copper carbonate as Catalyst
0	8.60	8.70
5	7.30	3.70
10	6.65	2.50
15	5.90	1.95
20	5.75	1.40
30	5.10	0.40
60	4.60	0.10

EXAMPLE 7

The oxygen scavenging activity of hydroxylammonium acid sulfate and N,N-diethylhydroxylamine over hydrazine is evident. The following compounds according to this invention show similar unexpected oxygen scavenging activities when tested by the procedure described in Example 1.

Example No.

- 8 Hydroxylamine phosphate
- 9 N-Ethylhydroxylamine
- 10 N,N-Dimethylhydroxylamine
- 11 O-Methylhydroxylamine
- 12 O-Hexylhydroxylamine
- 13 N-Heptylhydroxylamine
- 14 N,N-Dipropylhydroxylamine
- 15 O-Methyl N,N-diethylhydroxylamine
- 16 N-Octylhydroxylamine
- 17 O-Ethyl N,N-dimethylhydroxylamine
- 18 N,N-Diethylhydroxylamine hydrochloride
- 19 **N-methyl** N-ethylhydroxylamine
- 20 O-Methylhydroxylamine phosphate
- 21 N-Butylhydroxylamine
- 22 N-Benzylhydroxylamine (.beta.-Benzylhydroxylamine)
- 23 O-Benzylhydroxylamine (.alpha.-Benzylhydroxylamine)
- 24 N,N-Diethylhydroxylamine acetate

The presently used hydroxylamines may be catalyzed using any of a number of well known catalyst used in sodium sulfite or hydrazine boiler water treatm

----- 3954643
 classes:1 510/305 1 510/283 1 510/292 1 510/306 1 510/307 1 510/308 1 510/316 1 510/324 1 510/351 1 510/352 1 510/355 1 516/126
 score: 627

keywords: water;propylene;acetic;aqueous solution;alkyl;organic;residue;corresponding;sulfone;ratio;solution;hydrogen;compounds;ammonium;aqueous;metal;oxide;acid;methyl;ethylene;concentrations;carboxylic;nitrogen;substituted;ingredients;derivatives;magnesium;tartaric;sulfuric;cationic;potassium;amides;stated;dimethyl;extent;consists;amines;substances;water-soluble;citric acid;citric;anionic;published;ethers;bath;waste;temperatures;formulations;aromatic;alkaline;substance;oxygen;

- acid, 12% by weight of a C.sub.20 fatty acid, and 48% by weight of a C.sub.22 fatty acid (iodine value 4) (Soap B), respectively.
 "PEM" is the sodium salt prepared from 1:1 copolymerizate of ethylene and maleic acid anhydride with a specific viscosity 0.29 (1% by weight dissolved in dimethyl formamide at room temperature).
 "PPM" is the sodium salt prepared from a 1:1 copolymerizate of **propylene** and maleic acid anhydride with a specific viscosity 0.30 (1% by weight dissolved in dimethyl formamide at room temperature).
 "ABS" is the salt of an **alkyl**benzenesulfonic acid with 10 to 15, substantially 11 to 13, carbon atoms in the **alkyl** chain, obtained by condensation of straight-chain olefins with benzene and sulfonation of the **alkyl**benzene thus formed.
 "Alkanesulfonate" is a sulfonate obtained from paraffins having 12 to 16 carbon atoms by way of sulfoxidation.
 "KA-sulfate" and "TA-sulfate" are the salts of sulfated, substantially saturated
 - ate" is a product containing about 10% of active oxygen of the approximate composition NaBO.sub.2.H.sub.2 O.sub.2.3H.sub.2 O.
 "NTA", "EDTA" and "HEDP" are the salts of nitrilotriacetic acid, ethylenediaminetetraacetic acid and hydroxyethanediphosphonic acid, respectively.
 "CMC" is the salt of carboxymethylcellulose.
 "Stilbene brightener" is a compound of Formula I on page 12, in which R.sub.1 represents an anilino **residue** and R.sub.2 represents a morpholino **residue**.
 "Pyrazoline brightener" is a compound of Formula III on page 13, in which R.sub.7 represents a sulfamoyl **residue** and R.sub.8 represents chlorine.
 In all the examples the amounts given relate to the pure substances. These substances may contain accompanying substances, depending on the preparation as, for example, water, sodium sulfate, sodium chloride, sodium **carbonate**, etc. Since such accompanying substances do not in any way impair the activity of the individual substances

----- 6479374
 classes:1 438/601 1 438/624 1 438/637 1 438/594 1 438/476 1 438/786
 score: 618

keywords: amount of water;water;silicon oxide;substrate;removing;polyethylene glycol;polyethylene;carbonate;propylene glycol;propylene;ethylene glycol;acetic;hydroxide;etching;aqueous solution;alkyl;organic;substrates;etch;photoresist;claimed;corresponding;tetramethylammonium;solution;hydrogen;silicon;coating;compounds;solvent;aqueous;metal;oxide;acid;methyl;acetate;ethyl;ethylene;glycol;nitrogen;hydroxyl;functional groups;Stirring;derivatives;oxalic;formic;hydrochloric acid;hydrochloric;sulfuric;potassium;dimethyl;evaporation;novel;satisfactory;silicon dioxide;dioxide;aluminum oxide;contamination;fluorine;vapor;nitric;dielectric constant;polymers;alkaline;chemically;dissolving;substance;protective;completion;problems;acidic;poor;tungsten;titanium nitride;nitride;titanium;aluminum;oxygen;

- onnect delay is greatly suppressed, as compared to the case of the conventional circuit structures.
 The circuit structure obtained by the method of the present invention can be very advantageously used for producing a multi-layer circuit board and a semi-conductor device.

BEST MODE FOR CARRYING OUT THE INVENTION

Hereinbelow, the present invention will be described in more detail with reference to the following Examples and Comparative Examples, but they should not be construed as limiting the scope of the present invention.

EXAMPLE 1

0.74 g of methyltriethoxysilane, 2.4 g of tetraethoxysilane, 0.68 g of

****polyethylene** glycol monomethacrylate** having a number average molecular weight of 360 and 0.34 g of ****polyethylene** glycol dimethacrylate** having a number average molecular weight of 540 were dissolved in a mixed solvent of 2.0 g of ****N-methyl**pyrrolidone** and 1.0 g of ****propylene glycol** methyl ether acetate**. To the resultant solution were added 0.

- ator layer to obtain a laminate structure as shown in FIG. 1 which has a ****photoresist**** layer having a thickness of 1.05 .mu.m. A test pattern image was transferred to the ****photoresist**** layer by means of a light exposure apparatus (trade name: i-line stepper FPA300i4, manufactured and sold by CANON INC., Japan). The resultant ****photoresist**** layer was developed using a 2.38% ****aqueous solution**** of tetramethylammonium ****hydroxide**** to remove portions of the ****photoresist**** layer which had been exposed, thereby obtaining a structure as shown in FIG. 2 which has, on the surface of the preliminary insulator layer, a ****photoresist**** image corresponding to the test pattern image. The configuration of the test pattern will be explained below.

The preliminary insulator layer (comprising the ****silicon oxide****-organic polymer composite thin film) having the obtained ****photoresist**** image was subjected to ****etching**** through the ****photoresist**** image as a mask (i.e., a pr

----- 6110451
classes:1 424/7016 1 424/7011 1 424/7015 1 424/7017 1 424/7019
score: 617

keywords: water;pyrrolidone;substrate;removing;polyethylene glycol;polyethylene;pyrrolidone;propylene glycol;propylene;ethylene glycol;acetic;triethanolamine;diethanolamine;monoethanolamine;ethanol;alkyl;organic;substrates;cleaning;claimed;ratio;solution;silicon;compounds;solvent;amine;ammonium;aqueous;metal;oxide;acid;methyl;acetate;ethyl;ethylene;glycol;ranges;carboxylic;nitrogen;substituted;chosen;ingredients;benefits;ambient;derivatives;succinic;phosphoric;sulfuric;silicone;amphoteric;nonionic;cationic;surfactant;vinyl alcohol;potassium;amides;N-alkyl;ratios;dimethyl;discloses;contacting;amines;environmental;satisfactory;recognized;synergistic;solubility;citric acid;citric;anionic;vapor;published;ammonium salt;quaternary ammonium;quaternary;phase;issued;polymers;formulation;formulations;hydrocarbon;aromatic;polymeric;frequently;neutralized;poor;oxygen;reacted;undesirable;

- discloses many other anionic as well as other surfactant types and is incorporated herein by reference in its entirety.
- Preferred anionic deterative surfactants for use in the present compositions include ammonium lauryl sulfate, ammonium laureth sulfate, triethylamine lauryl sulfate, triethylamine laureth sulfate, ****triethanolamine**** lauryl sulfate, ****triethanolamine**** laureth sulfate, ****monoethanolamine**** lauryl sulfate, ****monoethanolamine**** laureth sulfate, ****diethanolamine**** lauryl sulfate, ****diethanolamine**** laureth sulfate, lauric monoglyceride sodium sulfate, sodium lauryl sulfate, sodium laureth sulfate, potassium lauryl sulfate, potassium laureth sulfate, sodium lauryl sarcosinate, sodium lauroyl sarcosinate, lauryl sarcosine, cocoyl sarcosine, ammonium cocoyl sulfate, ammonium lauroyl sulfate, sodium cocoyl sulfate, sodium lauroyl sulfate, potassium cocoyl sulfate, potassium lauryl sulfate, triethanolamine 1 lauryl sulfate, triethanol
- kyltaurines such as the one prepared by reacting dodecylamine with sodium isethionate according to the teaching of U.S. Pat. No. 2,658,072, N-higher ****alkyl**** aspartic acids such as those produced according to the teaching of U.S. Pat. No. 2,438,091, and the products sold under the trade name "MIRANOL".TM. as described in U.S. Pat. No. 2,528,378.

Optional Deterative Surfactants

In addition to the anionic deterative surfactant component, the compositions of the present invention can optionally contain other deterative surfactants. These include nonionic surfactants, and zwitterionic surfactants. Optional deterative surfactants, when used, are typically present at levels of from about 0.5% to about 20%, more typically from about 1% to about 10%, although higher or lower levels can be used. The total amount of deterative surfactant in compositions containing optional deterative surfactants in addition to the anionic surfactant will generally

- used in the compositions hereof.
- Nonionic deterative surfactants which can be used include those broadly defined as compounds produced by the condensation of ****alkyl****ene oxide groups (hydrophilic in nature) with an ****organic**** hydrophobic compound, which may be aliphatic or ****alkyl**** aromatic in nature. Examples of preferred classes of nonionic deterative surfactants are: The long chain alkanolamides; the ****polyethylene**** oxide condensates of ****alkyl**** phenols; the condensation product

of aliphatic alcohols having from about 8 to about 18 carbon atoms, in either straight chain or branched chain configuration, with ethylene oxide; the long chain tertiary amine oxides; the long chain tertiary phosphine oxides; the long chain dialkyl sulfoxides containing one short chain ****alkyl**** or hydroxy ****alkyl**** radical of from about 1 to about 3 carbon atoms; and the ****alkyl**** polysaccharide (APS) surfactants such as the ****alkyl**** polyglycosides; the ****polyethylene**** glycol (PEG)

----- 6495573

classes:1 514/342 1 514/340 1 514/365 1 514/370
score: 585

keywords: water;substrate;polyethylene;propylene;triethanolamine;diethanolamine;monoethanolamine;alkyl;organic;claimed;corresponding;independently;ratio;solution;hydrogen;silicon;compounds;solvent;aqueous;oxide;acid;methyl;acetate;ethyl;ethylene;glycol;concentrations;carboxylic;hydroxyl groups;nitrogen;carbonyl;hydroxyl;substituted;wetting;derivatives;magnesium;succinic;hydrochloric;phosphoric;silicone;classified;amides;N-alkyl;dimethyl;glycols;discloses;ammonia;contacting;resistant;substances;satisfactory;silicon dioxide;dioxide;adding;water-soluble;fluorine;published;ethers;ketones;phase;hydrophilic;Films;polymers;formulation;temperatures;formulations;hydrocarbon;aromatic;dissolving;substance;polymeric;oxygen;interaction;undesirable;

- -1,2- and -1,3-glycol, butylene-1,4- and -2,3-glycol, pentane-1,5-diol, hexane-1,6-diol, octane-1,8-diol, neopentyl glycol, 1,4-bis-hydroxymethylcyclohexane, 2-methyl-1,3-propanediol, glycerol, trimethylolpropane, hexane-1,2,6-triol, trimethylolethane, pentaerythritol, quinitol, mannitol and sorbitol, diethylene glycol, triethylene glycol, tetraethylene glycol, ****polyethylene**** glycols having a molecular weight of up to 400, dipropylene glycol, polypropylene glycols having a molecular weight of up to 400, dibutylene glycol, polybutylene glycols having a molecular weight of up to 400, 4,4'-dihydroxy-diphenylpropane, di-hydroxymethylhydroquinone, ****ethanol****amine, ****diethanolamine****, ****triethanolamine****, 3-aminopropanol, ethylenediamine, 1,3-diaminopropane, 1-mercapto-3-aminopropane, 4-hydroxy- or -amino-phthalic acid, succinic acid, adipic acid, hydrazine, N,N'-dimethylhydrazine, 4,4'-diaminodiphenylmethane, toluenediamine, methyl

----- 6500599

classes:1 430/2731 1 430/302
score: 569

keywords: water;pyrrolidone;acetic acid;methyl lactate;removing;polyethylene glycol;polyethylene;pyrrolidone;carbonate;propylene glycol;propylene;methyl lactate;lactate;acetic acid;acetic;hydroxide;aqueous solution;ethanol;alkyl;organic;substrates;residue;claimed;corresponding;solution;hydrogen;phosphonium;silicon;coating;compounds;solvent;amine;aqueous;metal;oxide;acid;methyl;acetate;ethyl;ethylene;glycol;carboxylic;ketone;hydroxyl groups;nitrogen;hydroxyl;functional groups;substituted;dissolution;derivatives;magnesium;tartaric;oxalic;malic;formic;hydrochloric;phosphoric;sulfuric;cationic;surfactant;desirably;discloses;ammonia;amines;novel;environmental;satisfactory;dioxide;water-soluble;fluorine;published;ketones;phase;hydrophilic;nitric acid;nitric;Films;polymers;waste;isopropanol;hydrocarbon;aromatic;alkaline;dissolving;protective;problems;acidic;poor;tungsten;titanium;aluminum;oxygen;

- dispersing agents are used as additives.

For improving the adhesion, well-known adhesion improvers (for example, silane coupling agents and titanate coupling agents) may be used.

In addition, various additives such as surfactants for improving coating properties are used as needed.

The compositions for the light-heat conversion layers used in the invention are dissolved or dispersed in appropriate solvents, and applied onto ****substrates**** and dried. Examples of the solvents include 2-methoxyethanol, 2-methoxyethyl acetate, ****propylene glycol**** methylethyl acetate, methyl ****lactate****, ****ethyl**** ****lactate****, ****propylene glycol**** monomethyl ether, ****ethanol****, isopropanol, methyl ethyl ketone, N,N-dimethylformamide, N,N-dimethylacetamide, tetrahydrofuran and dioxane. They may be used either alone or as a mixed solvent thereof.

The weight thereof coated is preferably from 0.5 g/m.² to 3 g/m.² by weight after drying. Less than 0.5 g/m.² gives unfavorable

res or aluminates.

As the cross-linking reaction used in the formation of the ****organic**** hydrophilic matrixes of the hydrophilic layers of the invention, the formation of covalent bonds by heat or light or the formation of ionic

bonds with multivalent metal salts is available.

As the **organic** hydrophilic polymers used in the invention, polymers having functional groups usable in the cross-linking reaction are preferred.

Preferred examples of the functional groups include --OH, --SH, --NH.sub.2, --NH--, --CO--NH.sub.2, --CO--NH--, --O--CO--NH--, --NH--CO--NH--, --CO--OH, --CO--O--, --CO--O.sup.-, --CS--OH, --CO--SH, --CS--SH, --SO.sub.3 H, --SO.sub.2 (O.sup.-), --PO.sub.3 H.sub.2, --PO(O.sup.31).sub.2, --SO.sub.2 --NH.sub.2, --SO.sub.2 --NH-- and the following functional groups.

##STR2##

In particular, hydroxyl, amino, carboxyl and epoxy groups are preferred.

As such **organic** hydrophilic polymers of the invention, well-known water-soluble

- e polymers can be used, and examples thereof include polyvinyl alcohol (polyvinyl acetate having a degree of saponification of 60% or more), modified polyvinyl alcohol such as carboxy-modified polyvinyl alcohol, starch and derivatives thereof, cellulose derivatives such as carboxymethyl cellulose, salts thereof and hydroxyethyl cellulose, casein, gelatin, gum arabic, polyvinyl-pyrrolidone, vinyl acetate-crotonic acid copolymers and salts thereof, styrene-maleic acid copolymers and salts thereof, polyacrylic acid and salts thereof, polymethacrylic acid and salts thereof, **polyethylene** glycol, **polyethylene**imine, polyvinylsulfonic acid and salts thereof, polyhydroxyethyl methacrylate, polyhydroxyethyl acrylate and polyacrylamide.

These polymers may be used either alone or as a combination of two or more of them. The amount thereof used is from 20% to 99% by weight, preferably from 25% to 95% by weight, and more preferably from 30%

- hydroxyl groups or alkoxyl groups at the same time to form resinous structures containing mixtures thereof. The polymers are in the sol state at a stage that there are many alkoxyl groups or hydroxyl groups, but the network resinous structures become strong with the progress of ether bonding. Further, they also have together the action that the hydroxyl groups are partly bonded to fine solid particles, thereby modifying the surfaces of the fine solid particles to change the hydrophilicity. Examples of the multivalent binding elements of compounds having the hydroxyl groups or alkoxyl groups performing the sol-gel conversion are aluminum, silicon, titanium and zirconium, which can be used in the invention. Sol-gel conversion systems due to siloxane bonds which can be most preferably used are described below. Sol-gel conversion using aluminum, titanium and zirconium can be conducted by replacing each element for silicon de

----- 6506937

classes:1 564/86 1 564/23 1 564/39 1 564/40 1 564/41 1 564/87 1 564/88 1 564/166 1 564/170 1 564/176 1 564/177 1 564/185 1 564/186

score: 568

keywords: sulfoxide;dms;removing;polyethylene;carbonate;ethylene glycol;acetic;hydroxide;ethanol;alkyl;organic;residue;claimed;sulfoxide;corresponding;solution;hydrogen;silicon;compounds;solvent;amine;aqueous;metal;acid;methyl;ethyl;ethylene;glycol;concentrations;substituted;wetting;derivatives;hydroxides;magnesium;tartaric;succinic;malonic;malic;benzoic;propionic;hydrochloric acid;hydrochloric;phosphoric;sulfuric;potassium;amides;stated;dimethyl sulfoxide;dimethyl;ammonia;extent;amines;metal ions;novel;substances;recognized;dioxide;adding;citric;fluorine;nitric;bath;temperatures;formulations;hydrocarbon;aromatic;alkaline;chemically;substance;protective;oxygen;reacted;undesirable;

- d in experimental physiology by 2-deoxyglucose by blockade of glucose meta-bolism. The action potential-shortening effect of these substances was prevented or reduced by the simultaneous dose of the test substances. The test substances were added to the bath solution as stock solutions in propanediol. The values stated relate to measurements 30 minutes after the addition. The APD.sub.95 in the presence of DEO or HOE 234 and in the absence of the test substance serves as a control.

(c) Results:

The following values were measured:

Measurement	APD.sub.95 -DEO.sup.a)	APD.sub.95 -HOE 234.sup.a)
	[ms]	[ms]
Control	<40	<40
Example 1	107 .+- 14 (155 .+- 9)	138 .+- 3 (160 .+- 20)
	n = 3	n = 3
Example 4	110 .+- 23 (180 .+- 5)	123 .+- 15 (172 .+- 18)
	n = 3	n = 3

Ex

- cell line, for some of the compounds according to the invention. The action strength of a compound in this model predicts the extent of the hypoglycemic potential of this compound.

(b) Method

Cell culture of RINm5F cells

RINm5F cells were cultured at 37.degree. C. in RPMI 1640 culture medium (flow), to which 11 mol of glucose, 10% (volume/volume) of fetal calf serum, 2 mmol of glutamine and 50 .mu.g/ml of gentamycin were added. For the studies, the cells were isolated by incubation (about 3 minutes) in a Ca.sup.2+ -free medium containing 0.25% of trypsin and stored on ice.

Measurement method

Isolated RINm5F cells were introduced into a Plexi-glas chamber on an inverse microscope fitted with a differential interference contrast lens. A fire-polished micropipette with an opening diameter of about 1 .mu.m was placed on the cell with the aid of a micromanipulator under optical control (400-fold magnification). By applying a slight reduced p

- he opener for ATP-sensitive K.sup.+ channels diazoxide (100 .mu.mol) was added to the bath solution in all the experiments. All the experiments were carried out at 34.+-.1.degree. C.

- (c) Results (The concentrations of the compounds according to the invention in the experiments are 10.sup.-5 mol per liter)

Measurement	.DELTA.U (mv).sup.a)
Example 1	13 (-76) n = 6
Example 4	19 (-76) n = 3
Example 10	11 (-79) n = 3

.sup.a) The measurement values from n experiments are followed by the corresponding blank values in parentheses. The blank values are the cell potentials under a dose of diazoxide.

EXAMPLE 19

2-Methoxy-5-chloro-N-(5[-1-sulfonylamino-N-(methyaminothiocarbonyl)-2-chlorophenyl]-ethyl)-benzamide

##STR34##

This compound was obtained in accordance with example 10 starting from 2-Methoxy-5-chloro-N-(5[-1-sulfonylamino-2-chlorophenyl]-ethyl)-benzamide

----- 6489080
 classes:1 430/2811 1 430/2701 1 430/2881 1 430/914
 score: 561

keywords: water;sulfoxide;ethanolamine;ethyl lactate;substrate;polyethylene;carbonate;propylene glycol;propylene;ethyl lactate;ethylene glycol;ethyl acetate;lactate;acetic;hydroxide;aqueous solution;alkyl;organic;claimed;sulfoxide;corresponding;tetramethylammonium;ratio;solution;hydrogen;phosphonium;silicon;coating;compounds;solvent;ammonium;aqueous;oxide;acid;methyl;acetate;ethyl;ethylene;glycol;ranges;carboxylic;hydroxyl groups;nitrogen;carbonyl;hydroxyl;substituted;dissolution;immersed;Stirring;derivatives;oxalic;formic;hydrochloric acid;hydrochloric;sulfuric;silicone;cationic;potassium;dimethyl;amines;substances;solubility;fluorine;European;published;ethers;phase;polymers;rinsed;aromatic;phenolic;alkaline;chemically;dissolving;protective;problems;acidic;neutralized;aluminum;tetramethylammonium hydroxide;oxygen;

- aminoaryl groups, arylamino groups, **alkyl** groups, alkoxy groups, acyl groups, acyloxy groups, aryl groups, aryloxy groups, nitro, hydroxy, and cyano. Specific examples of the most preferred **organic** basic compounds include guanidine, 1,1-dimethylguanidine, 1,1,3,3-tetramethylguanidine, 2-aminopyridine, 3-aminopyridine, 4-aminopyridine, 2-dimethylaminopyridine, 4-dimethylaminopyridine, 2-diethylaminopyridine, 2-(aminomethyl)pyridine: 2-amino-3-methylpyridine, 2-amino-4-methylpyridine, 2-amino-5-methylpyridine, 2-amino-6-methylpyridine, 3-aminoethylpyridine, 4-aminoethylpyridine, 3-aminopyrrolidine, piperazine, N-(2-aminoethyl)piperazine, N-(2-aminoethyl)piperidine, 4-amino-2,6,6-tetramethylpiperidine, 4-piperidinopiperidine, 2-iminopiperidine, 1-(2-aminoethyl)pyrrolidine, pyrazole, 3-amino-5-methylpyrazole, 5-amino-3-methyl-1-p-tolylpyrazole, pyrazine, 2-(aminomethyl)-5-methylpyrazine, pyrimidine, 2,4-diaminopyrimidine
- ereof exceeding 10 parts by weight tend to result in reduced sensitivity and impaired development resistance of unexposed areas.

The above-described components of the composition of the present invention are dissolved in a solvent and coated on a **substrate**. Examples of solvents preferably used in the present invention include ethylene dichloride,

cyclohexanone, cyclopentanone, 2-heptanone, .gamma.-butyrolactone, methyl ethyl ketone, **ethylene glycol** monomethyl ether, **ethylene glycol** monoethyl ether, 2-methoxyethyl acetate, **ethylene glycol** monoethyl ether acetate, **propylene glycol** monomethyl ether, **propylene glycol** monomethyl ether acetate, toluene, **ethyl acetate**, **methyl lactate**, **ethyl lactate**, methylmethoxy propionate, ethylethoxy propionate, methyl pyruvate, ethyl pyruvate, propyl pyruvate, N,N-dimethylformamide, dimethyl sulfoxide, **N-methylpyrrolidone, and tetrahydrofuran. These solvents can be used alone or in combinati

----- 6475700
classes:1 430/2781 1 430/2841
score: 561

keywords: amount of water;water;hydrofluoric;silicon oxide;removing;polyethylene glycol;polyethylene;propylene glycol;propylene;ethylene glycol;acetic;hydroxide;etching;aqueous solution;alkyl;organic;residue;claimed;corresponding;tetramethylammonium;ratio;solution;hydrogen;silicon;coating;compounds;solvent;amine;ammonium;aqueous;metal;oxide;acid;methyl;acetate;ethyl;ethylene;glycol;diacetate;ranges;carboxylic;ketone;hydroxyl groups;nitrogen;carbonyl;hydroxyl;functional groups;substituted;chosen;balance;immersed;Stirring;magnesium;tartaric;oxalic;malic;propionic;hydrochloric acid;hydrochloric;phosphoric;sulfuric;silicone;nonionic;cationic;vinyl alcohol;improves;zinc;potassium;amides;listed;dimethyl;desirably;ammonia;resistant;metal ions;inhibit;substances;adding;water-soluble;European;published;quaternary ammonium;quaternary;ketones;phase;hydrophilic;nitric acid;nitric;polymers;formulation;bath;waste;temperatures;aromatic hydrocarbon;hydrocarbon;aromatic;alkaline;chemically;protective;completion;acidic;titanium;aluminum;oxygen;species;

- hylamine, triethylamine, monoisopropylamine, diisopropylamine, triisopropylamine; n-butylamine, **monoethanolamine**, **diethanolamine**, triethanolamine, monoisopropanolamine, diisopropanolamine, ethyleneimine, ethylenediamine, and pyridine. These alkalis can be used either individually or as a combination of two or more thereof.

Among **aqueous solution**s of the above alkalis, an **aqueous solution** of an alkali metal silicate having a pH of 12 or higher particularly allows the effects of the invention be manifested. The developing properties of an alkali metal silicate **aqueous solution** are controllable by the ratio of the **silicon oxide** (SiO.sub.2) of the silicate to the alkali metal oxide (M.sub.2 O, generally represented in terms of SiO.sub.2 /M.sub.2 O molar ratio, and the concentration. From this point of view, it is preferable to use a sodium silicate **aqueous solution** having an SiO.sub.2 /Na.sub.2 O molar ratio of 1.0 to 1.5 a

----- 4824763
classes:1 430/258 1 430/326 1 430/329 1 430/330 1 430/331 1 510/176 1 510/407 1 510/499
score: 557

keywords: deionized water;water;pyrrolidone;substrate;removing;N-methyl pyrrolidone;pyrrolidone;N-methyl;organic;substrates;photoresist;stripping;claimed;corresponding;independently;solution;silicon;compounds;solvent;amine;aqueous;metal;acid;glycol;carboxylic;substituted;photoresist stripper;immersed;derivatives;sulfuric;deionized;stated;dimethyl;contacting;elevated;photoresist stripping;novel;substances;integrated circuits;integrated circuit;dioxide;solubility;contamination;bath;coatings;temperatures;aromatic hydrocarbon;hydrocarbon;aromatic;phenolic;alkaline;dissolving;polymeric;protective;acidic;nitride;aluminum;oxygen plasma;oxygen;species;

- ist easier to remove.

These and related objects may be achieved through use of the novel **stripping** solution and process for **stripping** positive **photoresist** and other difficult to remove polymeric coatings from **substrates** herein disclosed. A positive **photoresist** in accordance with this invention has a triamine as its principal active ingredient.

The **stripping** composition should contain from about 25 weight percent to about 100 weight percent of the triamine and from about 0 weight percent to about 75 weight percent of a suitable polar or nonpolar **organic** solvent.

The process for **stripping** positive **photoresist** from a **substrate** in accordance with this invention comprises contacting the positive **photoresist** with the above triamine composition at a temperature and time sufficient to remove the **photoresist**, typically at a temperature of about 80.degree. C. to about 120.degree. C. for a time of from about one minute to about 20 minute

Triamines which are useful in the composition and process of this invention have the general formula

##STR1##

wherein R.sub.1 and R.sub.2 are, independently in each case, a difunctional straight or branched chain hydrocarbyl or substituted hydrocarbyl group having from about 2 to about 20 carbon atoms, including straight and branched chain, heterocyclic, aromatic, halogen, hydroxy and cyano substituted aliphatic groups. Suitable specific examples of operable triamines for the composition and process of this invention include diethylene triamine, 1 benzyl- and 1 cyanoethyl- substituted diethylene triamine, 1,2 dibenzyl diethylene triamine, lauryl diethylene triamine, N-(2 hydroxyethyl) diethylene triamine, N-(2 hydroxypropyl) diethylene triamine, and the like. The preferred triamine is diethylene triamine.

Suitable specific examples of polar **organic** solvents for the composition and process include **N-methyl pyrrolidone**, di

----- 6506537
classes:1 430/2701 1 326/905 1 326/910
score: 534

keywords: water;ethanolamine;substrate;polyethylene glycol;polyethylene;propylene glycol;propylene;ethylene glycol;lactate;hydroxide;etching;diethanolamine;ethanol;alkyl;organic;substrates;claimed;corresponding;sulfone;independently;tetramethylammonium;solution;hydrogen;phosphonium;silicon;coating;compounds;solvent;ammonium;aqueous;metal;acid;methyl;ethyl;ethylene;glycol;ketone;nitrogen;carbonyl;hydroxyl;functional groups;substituted;ingredients;amphoteric;nonionic;cationic;stated;dimethyl;ammonia;amines;water-soluble organic;water-soluble;anionic;ketones;hydrophilic;Films;polymers;formulation;compositions used;hydrocarbon;aromatic;phenolic;alkaline;chemically;dissolving;acidic;tetramethylammonium hydroxide;alkanolamines;

- based on 100 parts by weight of the total resin components in the radiation-sensitive resin composition.

A dye and/or a pigment may also be mixed so that latent images at exposed areas can be rendered visible and any influence of halation can be lessened at the time of exposure, and an adhesion aid may be mixed so as to improve adhesion to **substrates**.

As other additives, they may further be halation preventive agents, storage stabilizers, anti-foaming agents and shape improvers, which may specifically include 4-hydroxy-4'-methylcalcon.

Solvents

The positive radiation-sensitive resin composition of the present invention is, when used, dissolved in a solvent so as to be in a solid concentration of, e.g., from 2 to 50% by weight, followed by filtration with a filter having a pore size of, e.g., about 0.2 .mu.m, and prepared into composition solutions.

The solvent may include, e.g., ethers, esters, ether esters, ketone esters, ketones

- glycol

di-n-propyl ether, diethylene glycol di-n-butyl ether, **ethylene glycol** monomethyl ether acetate, **ethylene glycol** monoethyl ether acetate, **propylene glycol** monomethyl ether acetate, **propylene glycol** monoethyl ether acetate, **propylene glycol** mono-n-propyl ether acetate, ethyl acetate, n-propyl acetate, n-butyl acetate, isopropenyl acetate, 3-methoxybutyl acetate, 3-methyl-3-methoxybutyl acetate, ethyl hydroxyacetate, ethyl ethoxyacetate, methyl acetoacetate, ethyl acetoacetate, isopropenyl propionate, 3-methyl-3-methoxybutyl propionate, **ethyl **lactate**, ethyl 2-hydroxy-2-methylpropionate, methyl 3-methoxypropionate, ethyl 3-methoxypropionate, methyl 3-ethoxypropionate, ethyl 3-ethoxypropionate, 3-methyl-3-methoxybutyl butyrate, methyl 2-hydroxy-3-methylbutyrate, methyl ethyl ketone, cyclohexanone, 2-heptane, 3-heptane, 4-heptane, N,N-dimethylformamide, N,N-dimethylacetamide, **N-methyl**pyrrolidone, toluene, and xyle

- aline aqueous

solutions prepared by dissolving at least one of alkaline compounds such as alkali metal **hydroxide**s, ammonia water, **alkyl**amines, alkanolamines, heterocyclic amines, tetraalkylammonium **hydroxide**s, choline, 1,8-diazabicyclo[5.4.0]-7-undecene and 1,5-diazabicyclo[4.3.0]-5-nonene so as to be in a concentration of usually from 1 to 10% by weight, and preferably from 2 to 5% by weight. Particularly preferred alkaline developing solutions are **aqueous solution**s of tetraalkylammonium **hydroxide**s.

To developing solutions formed of such alkaline **aqueous solution**s, water-soluble **organic** solvents or surface-active agents as exemplified by

methanol and **ethanol** may also be added in appropriate quantity. Incidentally, when such developing solutions formed of alkaline aqueous solutions are used, it is common to carry out water washing after the development. In the production of devices, the lower-layer **organic** films are

----- 4549968

classes:1 210/750 1 252/178 1 252/18828 1 252/392 1 252/393 1 422/13 1 422/16 1 422/17
score: 522

keywords: water;hydroxide;aqueous solution;residue;ratio;solution;amine;aqueous;acid;severe;ambient;state
d;amines;dioxide;adding;toxic;formulations;alkaline;substance;problems;oxygen;

- inbelow for about five months. The procedure consisted of taking the stored samples and first vigorously shaking the sample bottles to ensure homogeneity and to loosen any **residue** which may have adhered to the sample bottle walls. A 20 mL aliquot of each sample was then drawn into a 30 mL syringe, which was subsequently fitted with a 25 mm membrane filter holder containing a 0.22 micron pore size membrane. The membranes were previously dried for about 2 hours at 100.degree. C., cooled in a desiccator, and then weighed. The 20.0 mL aliquots were filtered through the membranes, yielding dark but clear filtrates. The clarity of the filtrates indicated that the 0.22 micron membranes were of sufficient pore size to quantitatively retain the sludge particles. The brown **residue**s obtained on the membrane filters were dried overnight in a desiccator over prepared silica gel (the **residue**s were not heated lest possibly losing the mo

----- 6506318

classes:1 252/70 1 106/13
score: 518

keywords: water;pyrrolidone;ethyl lactate;pyrrolidone;carbonate;propylene glycol;propylene;ethyl lactate;ethylene glycol;lactate;formate;ethanol;alkyl;organic;substrates;residue;claimed;ratio;solution;compounds;ammonium;aqueous;metal;acid;methyl;acetate;ethyl;ethylene;glycol;concentrations;carboxylic;nitrogen;hydroxyl;magnesium;succinic;malonic;potassium;nitrate;effectiveness;glycols;discloses;liquid phase;novel;environmental;dioxide;adding;teaches;solubility;contamination;spraying;carboxylate;phase;issued;formulation;batch;waste;toxic;temperatures;formulations;alkaline;chemically;problems;oxygen;species;

- /30% by weight **ethylene glycol** solution has a melting point of -18.degree. C.

EXAMPLE 42

The addition of 50% by weight of a 50% mixture of **ethyl **lactate** in water to a concentrated, filtered corn steep liquor (containing 50% water and 50% solids comprising mostly lactic acid and sugars) caused a reduction in freezing point from -11.degree. C. to -16.degree. C. The addition of 2% by weight sodium **lactate** further reduced the freezing point to -20.degree. C.

EXAMPLE 43

A mixture of 60% by weight water, 20% by weight sodium **lactate**, 2% by weight proline (an amino acid), 8% by weight sorbitol and 10% by weight sodium **pyrrolidone** carboxylate (sodium PCA) was prepared. No crystal formation at -35.degree. C. was observed. The pH was 6.57. For comparison a 50% by weight solution **propylene glycol** has a freezing point of -36.degree. C.

EXAMPLE 44

A mixture of 12% by weight **methyl **lactate**, 44% by weight methyl glucoside and 44% by weight water was prepared. A melting point of -18.degree. C. was observed. The mixture had a pH of 5.

EXAMPLE 45

A mixture containing 35% by weight **methyl **lactate**, 35% by weight methyl glucoside and 30% by weight water has a melting point of -21.degree. C. as determined by DSC.

EXAMPLE 46

A filtered concentrated liquid **residue** of a 50% mixture of corn stillage and steepwater containing 50% by weight water with a freezing point of -12.degree. C. is heated to 90.degree. C. and treated with 5% **ethanol** for 8 hours. The resulting mixture has a freezing point of -17.degree. C. The addition of 2% sodium **lactate** further reduces the freezing point to -21.degree. C.

EXAMPLES 47-53

Additional deicing and anti-icing fluids containing potassium **carbonate** were prepared in accordance with the present invention. The results are set forth below in Table 3.

TABLE 3

Example	47	48	49	50	51	52	53
Composition							
Methyl Glucosid							

----- 6492086

classes:1 430/2701 1 430/905 1 430/910

score: 515

keywords: water;ethyl lactate;substrate;propylene glycol;propylene;ethyl lactate;ethylene glycol;ethyl acetate;lactate;hydroxide;etching;aqueous solution;alkyl;organic;substrates;photoresist;stripping;claimed;corresponding;independently;tetramethylammonium;ratio;solution;hydrogen;tetrabutyl;silicon;coating;compounds;solvent;amine;ammonium;aqueous;acid;methyl;acetate;ethyl;ethylene;glycol;concentrations;ester groups;nitrogen;hydroxyl;substituted;dissolution;balance;Stirring;cationic;surfactant;listed;stated;amines;novel;recognized;integrated circuit;silicon dioxide;dioxide;solubility;European;quaternary ammonium;quaternary;phase;ammonium hydroxide;Films;polymers;formulation;bath;isopropanol;temperatures;formulations;hydrocarbon;aromatic;phenolic;alkaline;chemically;dissolving;poor;aluminum;involving;tetramethylammonium hydroxide;oxygen;reacted;interaction;

- position of the invention can be prepared by dissolving the components of the **photoresist** in a suitable solvent such as, for example, **ethyl **lactate**, **ethylene glycol** monomethyl ether, **ethylene glycol** monomethyl ether acetate, **propylene glycol** monomethyl ether; **propylene glycol** monomethyl ether acetate and 3-ethoxyethyl propionate. Typically, the solids content of the composition varies between about 5 and 35 percent by weight of the total weight of the **photoresist** composition. The resin binder and photoactive components should be present in amounts sufficient to provide a film coating layer and formation of good quality latent and relief images. See the examples which follow for exemplary preferred amounts of resist components.
- The compositions of the invention are used in accordance with generally known procedures. The liquid coating compositions of the invention are applied to a **substrate** such as by spinning, dipping, roller coating.
- photoresist coating over the **substrate**, the developed **substrate** may be selectively processed on those areas bared of resist, for example by chemically **etching** or plating **substrate** areas bared of resist in accordance with procedures known in the art. For the manufacture of microelectronic **substrates**, e.g., the manufacture of silicon dioxide wafers, suitable etchants include a gas etchant, e.g. a halogen plasma etchant such as a chlorine or fluorine-based etchant such as Cl.sub.2 or CF.sub.4 /CHF.sub.3 etchant applied as a plasma stream. After such processing, resist may be removed from the processed **substrate** using known **stripping** procedures.

All documents mentioned herein are incorporated herein by reference. The following non-limiting examples are illustrative of the invention.

EXAMPLES 1-6

Syntheses of Cage and Lactone Monomers

EXAMPLE 1

Synthesis of Ethyl Fenchol Methacrylate

##STR11##

Materi	Amount
--------	--------

----- 6506759

classes:1 514/255 1 514/365 1 514/419 1 514/415 1 514/428 1 514/392 1 514/307 1 546/87 1 546/85 1 546/146 1 546/147 1 546/150 1 548/146 1 548/452 1 548/494 1 548/495 1 548/567 1 548/569 1 548/566 1 544/402

score: 509

keywords: water;sulfoxide;dmsO;acetic acid;substrate;polyethylene;carbonate;ethylene glycol;ethyl acetate;lactate;acetic acid;acetic;hydroxide;aqueous solution;ethanol;alkyl;organic;residue;claimed;sulfoxide;corresponding;sulfone;independently;solution;hydrogen;compounds;solvent;amine;ammonium;aqueous;metal;acid;methyl;acetate;ethyl;ethylene;glycol;concentrations;carboxylic;ketone;nitrogen;carbonyl;hydroxyl;substituted;wetting;ambient;Stirring;derivatives;magnesium;succinic;oxalic;benzoic;propionic;formic;organic acids;hydrochloric acid;hydrochloric;phosphoric;classified;potassium;listed;dimethyl;effectiveness;glycols;discloses;ammonia;evaporation;amines;inhibit;novel;substances;recognized;adding;fluorinated;citric acid;citric;carboxylate;ammonium salt;ethers;phase;issued;bath;isopropanol;formulations;hydrocarbon;aromatic;alkali

ne;aluminum;involves;oxygen;reacted;

----- 6482567
 classes:1 430/2701 1 430/920 1 430/921 1 540/460 1 540/461 1 540/477 1 548/420 1 548/425 1 548/452 1 546/
 183 1 568/30 1 568/31 1 568/34
 score: 473

keywords: hydrofluoric;ethyl lactate;substrate;propylene glycol;propylene;ethyl lactate;ethylene glycol;lactate;hydroxide;etching;alkyl;substrates;etch;photoresist;claimed;corresponding;independently;hydroxylamine;solution;silicon;coating;compounds;solvent;amine;ammonium;aqueous;oxide;acid;methyl;acetate;ethyl;ethylene;glycol;concentrations;ester groups;ketone;hydroxyl groups;carbonyl;hydroxyl;functional groups;substituted;dissolution;malonic;cationic;nitrate;stated;consists;amines;novel;silicon dioxide;dioxide;vapor;European;published;Films;dimensions;polymers;formulation;temperatures;aromatic hydrocarbon;hydrocarbon;aromatic;phenolic;alkaline;chemically;dissolving;involving;oxygen plasma;oxygen;reacted;undesirable;

- minum-aluminum oxide microelectronic wafer.
 Gallium arsenide, ceramic, quartz or copper **substrates** may also be employed. Printed circuit board **substrates** such as copper clad laminates are also particularly preferred. The **photoresist**s of the invention will be particularly useful for circuit board imaging, including through hole and other aperture plating. Typical printed circuit board **substrates** have one or more copper layers interleaved with resin layers, such as epoxy layers. Substrates used for liquid crystal display and other flat panel display applications are also suitably employed, e.g. glass **substrates**, indium tin oxide coated **substrates** and the like.

A liquid coating resist composition may be applied by any standard means such as spinning, dipping or roller coating. **photoresist**s of the invention also may be formulated and applied as dry film resists, particularly for printed circuit board manufacture applications. The expos

- ed of
 photoresist in accordance with procedures known in the art. Suitable etchants include a hydrofluoric acid **etching** solution and a plasma gas etch such as an oxygen plasma etch.

All documents mentioned herein are incorporated herein by reference. The following non-limiting examples are illustrative of the invention.

EXAMPLE 1

A PAG of the invention of the structure NC(1-naphthyl)C.dbd.NOS(O).sub.2 C.sub.6 F.sub.5 can be prepared by reaction NCCH.sub.2 --(1-naphthyl) with amyl nitrate to provide the oxime NCCH(.dbd.NOH) (1-naphthyl). That oxime is reacted with pentafluorobenzenesulfonyl chloride to provide NC(1-naphthyl)C.dbd.NOS(O).sub.2 C.sub.6 F.sub.5.

EXAMPLE 2

Photoresist Preparation and Lithographic Processing

A **photoresist** of the invention is prepared by mixing the following components with amounts expressed as weight percent based on total weight of the resist compositions:

Resist components	Amount
-------------------	--------

----- 6479211
 classes:1 430/2701 1 430/905
 score: 451

keywords: water;dimethylsulfoxide;propylene carbonate;ethyl lactate;acetic acid;substrate;propylene carbonate;carbonate;propylene glycol;propylene;ethyl lactate;ethylene glycol;ethyl acetate;lactate;acetic acid;acetic;hydroxide;etching;aqueous solution;alkyl;organic;residue;photoresist;claimed;corresponding;independently;dimethylsulfoxide;tetramethylammonium;ratio;solution;hydrogen;phosphonium;silicon;coating;compounds;solvent;amine;ammonium;aqueous;oxide;acid;methyl;acetate;ethyl;ethylene;glycol;carboxylic;ketone;nitrogen;carbonyl;hydroxyl;functional groups;substituted;dissolution;Stirring;derivatives;hydrochloric acid;hydrochloric;sulfuric;nonionic;classified;surfactant;potassium;dimethyl;butyl acetate;discloses;evaporation;elevated;amines;integrated circuits;satisfactory;integrated circuit;dioxide;solubility;aluminum oxide;fluorine;introducing;European;published;ammonium salt;quaternary ammonium;quaternary;ethers;hydrophilic;bath;rinsed;hydrocarbon;aromatic;alkaline;dissolving;protective;completion;problems;neutralized;titanium;aluminum;tetramethylammonium hydroxide;oxygen;reacted;

S1/S2	(70/30)	W-2			
2	25	6 nm	1.1		
25g	1	(25)	S1/S2/S5	(70/27/3)	W-3
1	20	5 nm	1.1		

				report09903064.txt	
26g	1	(26)	S1/S2	(70/30)	W-1
3	40	6 nm	0.7		
Comparative					
Example					
1g	1	(R1)	S1/S2	(80/20)	none
3	15000	18 nm	0.1		
2g	1	(1)	S1/S2	(80/20)	W-4
1	300	12 nm	0.4		

As apparent from the results in Table 9, the samples of Comparative Examples have problems in the number of development failures and the generation of edge roughness. On the other hand, the positive **photoresist** compositions for far ultraviolet exposure of the present invention are excellent in the sensitivity, resolution, adhesion to a **substrate** and dry **etching** resistance and further pitch.

EXAMPLES 1h TO 26h AND COMPARATIVE EXAMPLES 1h AND 2h

1.4 g of a resin shown in Table 10, which was synthesized in Synthesis Examples above, 0.18 g of a photo-acid generator, 11.4 g of solvents (at a mixing ratio shown in Table 10), a surfactant (added in an amount of 1 wt % based on all solid contents in the composition) and 10 mg of an **organic** basic compound were mixed as shown in Table 10 and filtered through a 0.1- μ m microfilter to prepare positive **photoresist** composition solutions of Examples 1h to 26h and Comparative Examples 1h and 2h.

In Examples and Comparative Examples, the photo-acid generator used was triphenylsulfonium triflate. Resin R1 used in Comparative Example of Table 10 was the resin used in Example 1 of JP-A-11-109632.

The solvents used were:

S1: **ethyl**lactate**
 S2: **propylene glycol** monomethyl ether acetate
 S3: γ -butyrolactone
 S4: **propylene**carbonate**
 S5: ethylene **carbonate**
 S6: butyl acetate
 S7: 2-heptanone
 The

----- 5646240

classes:1 528/403 1 524/604 1 524/722 1 524/729 1 524/755 1 524/765 1 524/770 1 524/777 1 524/779 1 524/829 1 525/472 1 525/509 1 525/510 1 525/515 1 528/230 1 528/248 1 528/253 1 528/254 1 528/258 1 528/393 1 528/421 1 528/422 1 528/423
 score: 446

keywords: amount of water;deionized water;water;hydrofluoric;sulfoxide;chelating;propylene carbonate;pyrrolidone;removing;polyethylene;pyrrolidone;propylene carbonate;carbonate;propylene glycol;propylene;ethylene glycol;ethyl acetate;hydroxide;chelating;aqueous solution;triethanolamine;ethanol;alkyl;organic;fluoride;substrates;residue;cleaning;sulfoxide;corresponding;hydroxylamine;hydrazine;ratio;solution;hydrogen;phosphonium;coating;compounds;solvent;amine;ammonium;aqueous;metal;oxide;acid;methyl;acetate;ethyl;ethylene glycol;concentrations;carboxylic;ketone;hydroxyl groups;nitrogen;hydroxyl;functional groups;substituted;chosen;dissolution;blend;benefits;stirring;derivatives;hydroxides;magnesium;succinic;oxalic;benzoic;propionic;formic;hydrochloric;phosphoric;sulfuric;silicone;nonionic;cationic;zinc nitrate;zinc;potassium;nitrate;polyols;amides;deionized;ratios;dimethyl sulfoxide;dimethyl;desirably;butyl acetate;ammonia;evaporation;extent;elevated;consists;amines;resistant;metal ions;novel;substances;satisfactory;dioxide;adding;fluorinated;synergistic;water-soluble;solubility;introducing;vapor;quaternary ammonium;quaternary;ethers;ketones;nitric;Films;polymers;formulation;waste;temperatures;aromatic hydrocarbon;hydrocarbon;aromatic;phenolic;alkaline;chemically;dissolving;substance;polymeric;protective;completion;problems;acidic;neutralized;poor;tungsten;nitride;titanium;aluminum;involves;oxygen;reacted;

- content of the modified amino resin, of pentane were added under vigorous stirring. Stirring was continued further for about four minutes to disperse the reaction mixture uniformly, whereby a thermosetting expansion-forming composition was obtained.

The composition was treated in a similar manner to Example 120, subjected to foaming at about 37.degree. C., which was the temperature of the composition at the time of foaming, and then cured, whereby an elastic foam was obtained. As a result of tests, the elastic foam so obtained was found to have 12 g/l bulk density and 21 mm deflection at break.

EXAMPLE 122

In a 2l flask equipped with a stirrer, a thermometer and a condenser, 300.3 g of paraformaldehyde (80% grade, 8.0 moles in terms of 100% formaldehyde), 170.0 g of deionized water and 100 g of **ethanol** were charged, followed by adjustment of pH to 13.0 with a 30% **aqueous solution** of caustic soda. To the resultant mixture, 604.7 g (

----- 6479039
 classes:1 424/61 1 424/407
 score: 434

keywords: water;removing;propylene glycol;propylene;ethylene glycol;alkyl;organic;claimed;corresponding;ratio;solution;coating;compounds;solvent;amine;ammonium;aqueous;acid;methyl;acetate;ethyl;ethylene;glycol;ranges;concentrations;beneficial;hydroxyl groups;hydroxyl;substituted;ingredients;benefits;derivatives;listed;dimethyl;butyl acetate;amines;resistant;inhibit;substances;satisfactory;dioxide;contamination;vapor;published;quaternary ammonium;quaternary;ketones;polymers;formulation;coatings;formulations;phenolic;chemically;polymeric;neutralized;titanium;aluminum;species;interaction;

- e emollients and mitigators include allantoin, its metallic salts, and **organic** conjoiners. Emollients and mitigators can be employed in amounts ranging between about 0.001-5.0 percent by weight, preferably between about 0.01-3.0 percent by weight, and more preferably between about 0.05-2.0 percent by weight.

One or more solvents can be used to facilitate suspension of solid components in the composition of the present invention. The solvents are preferably non-aqueous. Such solvents are well known to those skilled in the art, and exemplary types of solvents compatible with the composition of the present invention include alkanes, alkanols, ketones, esters including acetates, amides, ethers, alcohols, glycol-ethers and nitroparaffins. Specific examples of these types of solvents include ethyl ether, petroleum ether, **methyl acetate**, acetone, cyclohexane, ethyl acetate, methyl ethyl ketone, carbon tetrachloride, ethyl alcohol, n-b

----- 5744504
 classes:1 521/50 1 428/365 1 428/3692 1 428/524 1 524/442 1 525/472 1 525/473 1 525/509 1 528/230 1 528/248 1 528/253 1 528/254
 score: 432

keywords: amount of water;deionized water;water;hydrofluoric;sulfoxide;chelating;propylene carbonate;pyrrolidone;removing;polyethylene;pyrrolidone;propylene carbonate;carbonate;propylene glycol;propylene;ethylene glycol;ethyl acetate;hydroxide;chelating;aqueous solution;triethanolamine;ethanol;alkyl;organic;fluoride;substrates;residue;cleaning;sulfoxide;corresponding;hydroxylamine;hydrazine;ratio;solution;hydrogen;phosphonium;coating;compounds;solvent;amine;ammonium;aqueous;metal;oxide;acid;methyl;acetate;ethyl;ethylene;glycol;concentrations;carboxylic;ketone;hydroxyl groups;nitrogen;hydroxyl;functional groups;substituted;chosen;dissolution;blend;benefits;Stirring;derivatives;hydroxides;magnesium;succinic;oxalic;benzoic;propionic;formic;hydrochloric;phosphoric;sulfuric;silicone;nonionic;cationic;zinc nitrate;zinc;potassium;nitrate;polyols;amides;deionized;ratios;dimethyl sulfoxide;dimethyl;desirably;butyl acetate;ammonia;evaporation;extent;elevated;consists;amines;resistant;metal ions;novel;substances;satisfactory;dioxide;adding;fluorinated;synergistic;water-soluble;solubility;introducing;vapor;quaternary ammonium;quaternary;ethers;ketones;nitric;Films;polymers;formulation;waste;temperatures;aromatic hydrocarbon;hydrocarbon;aromatic;phenolic;alkaline;chemically;dissolving;substance;polymeric;protective;completion;problems;acidic;neutralized;poor;tungsten;nitride;titanium;aluminum;involves;oxygen;reacted;

- ith a 20% **aqueous solution** of caustic soda. To the resultant mixture, 604.7 g (2.0 moles) of the diguanamine >formula (2)! prepared in the same manner as in Example 3 were added and mixed. The resulting mixture was reacted at 70.degree. C. for 1 hour while its pH was maintained at 10.0-10.5.

In the next place, 41.6 g (0.4 mole) of sodium bisulfite were added to the reaction mixture and while maintaining its pH at 10.0-10.5, a reaction was conducted at 80.degree. C. for 2 hours. The reaction mixture was adjusted to pH 7.0 with a 20% **aqueous solution** of sulfuric acid, whereby a modified amino resin was obtained.

Deionized water was added to the modified amino resin to adjust the solid resin content of the resulting solution to 74.0 wt. %. To the resulting mixture, 3.0 wt. % and 1.5 wt. %, each based on the solid content of the modified amino resin, of formic acid and sodium **alkyl**sulfonate ("Ratamur PS", trade name; 40% grade, produ

----- 6491970
 classes:1 427/130 1 427/131 1 427/132 1 427/289 1 427/404 1 427/4071 1 427/420 1 427/4342
 score: 427

keywords: water;substrate;removing;polyethylene;propylene;alkyl;organic;substrates;cleaning;claimed;corresponding;sub;independently;ratio;solution;hydrogen;coating;compounds;solvent;ammonium;metal;oxide;acid;me

thyl;acetate;ethyl;ethylene;glycol;ranges;concentrations;carboxylic;ketone;beneficial;hydroxyl groups;nitrogen;hydroxyl;substituted;blend;wetting;ambient;phosphoric;silicone;vinyl alcohol;improves;zinc;polyols;listed;desirably;effectiveness;evaporation;contacting;substances;satisfactory;adding;fluorinated;vapor;quaternary ammonium;quaternary;ethers;ketones;Films;polymers;formulation;coatings;formulations;hydrocarbon;aromatic;phenolic;dissolving;polymeric;problems;acidic;titanium;involves;reacted;

- rmance of the agent if E and A were to be ortho to each other.
- Preferably, the plurality of magnetic particles are first prepared as a concentrated magnetic particle dispersion prior to its addition to the binder. The concentrated magnetic particle dispersion can be prepared by procedures known to those in the dispersion art. The dispersion can be prepared by the use of a dispersing machine, for example, a high speed impeller mill, an attritor, or a sand mill.
- The concentrated magnetic particle dispersion can be diluted with a suitable non-aqueous ****organic**** solvent to make a magnetic coating composition. Typically, the non-aqueous ****organic**** solvent has dissolved or dispersed therein a binder, as described above. Solvents useful for dilution of the concentrated magnetic dispersion include ketones such as acetone, methyl ethyl ketone, methyl isobutyl ketone, cyclohexanone, isophorone; esters such as ****methyl acetate****, ****ethyl acetate****, bu

----- 6482848
 classes:1 514/418 1 548/467 1 548/468 1 548/486
 score: 426

keywords: water;dms0;dimethylsulfoxide;co-solvent;substrate;removing;polyethylene glycol;polyethylene;propylene;ethyl acetate;acetic;co-solvent;hydroxide;alkyl;organic;substrates;residue;claimed;corresponding;independently;dimethylsulfoxide;ratio;solution;hydrogen;coating;compounds;solvent;amine;ammonium;aqueous;metal;acid;methyl;acetate;ethyl;glycol;concentrations;carboxylic;beneficial;nitrogen;carbonyl;substituted;chosen;severe;ingredients;ambient;derivatives;hydroxides;magnesium;tartaric;succinic;malic;formic;organic acids;hydrochloric acid;hydrochloric;phosphoric;sulfuric;surfactant;zinc;potassium;deionized;listed;N-alkyl;stated;dimethyl;effectiveness;extent;contacting;consists;amines;inhibit;novel;substances;adding;solubility;citric acid;citric;European;published;quaternary ammonium;quaternary;phase;nitric;polymers;formulation;bath;coatings;temperatures;formulations;hydrocarbon;aromatic;substance;polymeric;acidic;titanium;involves;involving;oxygen;species;reacted;interaction;

- tisol Based: This formulation uses Captisol and an acidic agent to form an in situ salt at a pH of 1.5-2.0 to compound and lyophilize solutions of drug at concentrations of 20.0-25.0 mg/mL. The lyophilized cake is reconstituted with an IV fluid to provide a stable infusate at 2 mg/mL or higher at pH 3.
- (b) Non-Captisol based: This formulation uses small amounts of a surfactant such as Polysorbate-80 or Cremophor EL and an acidic agent to form an in-situ salt at a pH of 1.5-2.0 to compound and lyophilize solutions of drug at concentrations of 20.0-25.0 mg/mL. The lyophilized cake is reconstituted with cosolvent-surfactant based aqueous diluent such as PEG-300-Polysorbate 80 or PEG-300-Cremophor EL to provide a stable infusate at 2 mg/mL or higher at pH 3.
- (2) Sterile API fill:
 The drug is filled as a sterile powder fill in a container and will be reconstituted with a specific ****co-solvent****---surfactant based aqueous diluent to prov
 ide a stable infusate at 2 mg/mL or higher of drug at pH 3.
- [B] Solution Concentrate to be Diluted to a Stable Infusate (Table 2)
 The drug is solubilized in a non-aqueous mixture of ****co-solvent****s and surfactants at a high concentration such that it can be diluted with aqueous diluents to a stable infusate. The concentration of the drug in the infusate is at a concentration of 2 mg/mL or higher, at pH 3. The total levels of the ****co-solvent**** is less than 15% and the levels of surfactant is than 0.5%.

TABLE 1

Formulation		(1) Solid
formulations		
Attributes	Lyophilized - Captisol based	Lyophilized Non-Captisol
based	Sterile API Fill	
Dose/50 CC	200-300	200-300
	300-400	
vial (mgs)		
Sterile API fill NA		NA
	300-400 mg	

in vial
Composition - drug (mg) 200-300 dru
tion - drug (mg/mL) 20-30 pH
3.0 pH 3.0
Prelyophilate Acid (Molar) 1.4 drug (mg/mL)
20-30 NA
1.4 Antioxidant (mg/mL) 0-10 Acid (Molar)
0-10 Captisol (mg/mL) 200-300 Antioxidant (mg/mL)
20-30 Water for Injection qs to 1.0 mL Filler (mg/mL)
pH 1.5-2.0 Polysorbate-80 (mg)
0-50 Water for Injection qs
to 1.0 mL
pH
1.5-2.0
Acids used: Methane sulfonic acid, Tartaric acid, Citric acid, succinic
acid is used in a 1:1.4 molar ratio for in situ salt formation
Cosolvents- PEG-300, PEG-400
Surfactants: Polysorbate-80, Cremophor EL
Captisol .RTM.: Sulfobutylether Cyclodextrin
Drug: Compound of Example 2

TABLE 2

Composition

----- 6506419
classes:1 424/732 1 424/52
score: 407

keywords: water;gallic;removing;polyethylene glycol;polyethylene;carbonate;propylene glycol;propylene;ace
tic;gallic acid;aqueous solution;ethanol;alkyl;organic;residue;claimed;independently;ratio;solution;galli
c;hydrogen;compounds;solvent;aqueous;acid;methyl;acetate;ethyl;glycol;ketone;hydroxyl;Stirring;derivative
s;nonionic;potassium;discloses;substances;adding;water-soluble;citric acid;citric;published;phase;hydroph
ilic;phenolic;substance;completion;oxygen;

- mposition 22) 94.41
Composition 22 99.00
Based on the above results, it is readily understood that the compositions
of the present invention provide excellent stability of proanthocyanidin
in comparison with the compositions of control groups.

REFERENCE EXAMPLE 1

Production of a Turbid Apple Juice

One ton of washed fruits of an apple variety "Fuji" were applied to a
crusher and an **aqueous solution** of 10 kg of 10% ascorbic acid was added
thereto. Next, the juice was squeezed using a press and applied to a
screen of 60 meshes. Thereafter, this was sterilized at 95.degree. C. for
20 seconds using a flash sterilization machine, immediately cooled and
then centrifuged to obtain the turbid apple juice.

REFERENCE EXAMPLE 2

Production of a Casked Grape Fermentation Liquid

Ten tons of fruits of a grape variety "Cabernet Sauvignon" were applied to
a crusher, mixed with 800 g of potassium pyrosulfite and then with

----- 6498163
classes:1 514/2641 1 514/2581 1 514/2641 1 544/279
score: 372

keywords: water;sulfoxide;dms;dimethylsulfoxide;substrate;carbonate;propylene glycol;propylene;ethylene
glycol;ethyl acetate;lactate;acetic;hydroxide;aqueous solution;ethanol;alkyl;organic;residue;claimed;sulf
oxide;corresponding;independently;dimethylsulfoxide;solution;hydrogen;compounds;solvent;amine;ammonium;aq
ueous;metal;oxide;acid;methyl;acetate;ethyl;ethylene;glycol;monolaurate;concentrations;carboxylic;nitroge
n;carbonyl;functional groups;substituted;Stirring;derivatives;magnesium;propionic;hydrochloric;potassium;
listed;stated;ammonia;contacting;elevated;amines;inhibit;substances;recognized;adding;solubility;aluminum
oxide;carboxylate;ethers;phase;ammonium hydroxide;formulation;bath;isopropanol;rinsed;temperatures;formu
lations;hydrocarbon;aromatic;dissolving;protective;acidic;neutralized;aluminum;reacted;interaction;

- ed to a suspension of insect cell lysis
buffer comprised of 150 mM NaCl, 50 mM Tris pH 7.5, 1 mM DTT, 1% NP-40, 2

mM EGTA, 1 mM sodium vanadate, 1 mM PMSF, 1 .mu.g/mL each of leupeptin, pepstatin, and aprotinin. Insect cell lysate containing c-Src protein was incubated with these beads for 3 to 4 hours at 4.degree. C. with rotation. At the end of the lysate incubation, the beads were rinsed three times in lysis buffer, resuspended in lysis buffer containing 10% glycerol, and frozen. These latex beads were thawed, rinsed three times in assay buffer (40 mM Tris, pH 7.5, 5 mM .mu.gCl.sub.2) and suspended in the same buffer. In a Millipore 96-well plate with a 0.65 .mu.m polyvinylidene membrane bottom were added the reaction components: 10 .mu.L c-Src beads, 10 .mu.L of 2.5 mg/mL poly GluTyr **substrate**, 5 .mu.M ATP containing 0.2 .mu.Ci labeled .sup.32 P-ATP, 5 .mu.L DMSO containing inhibitors or as a solvent control, and buff

- ally have been shown to be bioavailable in animals, reaching peak plasma levels in nude mice in the range of about 10 nM to about 200 nM within 30 minutes following oral dosing at levels of about 4 to 5 mg/kg as suspensions in **lactate** buffer solutions having pH of 4.0. For example, the compound of Example 60 was administered orally at 5 mg/kg to mice, and plasma levels of about 200 nM were measured at 30 minutes following dosing. The compound was also administered intraperitoneally at 12 mg/kg and produced a peak plasma concentration of 10,000 nM at 30 minutes following dosing. When evaluated in female nude mice bearing subcutaneous MCF-7 human mammary tumor xenografts, the compound of Example 60 showed statistically insignificant tumor growth inhibitions at doses of 5 to 20 mg/kg when dosed on a schedule of q12 h.times.2; days 1-14.

The invention compounds can be formulated in conventional manners to provide convenient d

----- 6506738
 classes:1 514/80 1 514/25406 1 514/269 1 514/322 1 514/363 1 514/364 1 514/370 1 514/381 1 514/387 1 544/
 139 1 544/310 1 544/370 1 546/199 1 546/2737 1 548/113 1 548/139 1 548/132 1 548/181 1 548/250 1 548/3507
 score: 355

keywords: water;co-solvent;ethanolamine;acetic acid;polyethylene;carbonate;ethyl acetate;acid methyl;acet
 ic acid;acetic;co-solvent;hydroxide;aqueous solution;formate;ethanol;alkyl;organic;fluoride;residue;claim
 ed;corresponding;sulfone;independently;hydroxylamine;hydrazine;solution;hydrogen;compounds;solvent;amine;
 ammonium;aqueous;oxide;acid;methyl;acetate;ethyl;nitrogen;hydroxyl;substituted;severe;ingredients;ambient
 ;Stirring;derivatives;magnesium;tartaric;hydrochloric acid;hydrochloric;surfactant;potassium;listed;dimet
 hyl;desirably;glycols;consists;amines;inhibit;dioxide;adding;fluorine;published;quaternary;phase;formulat
 ion;bath;isopropanol;formulations;aromatic;dissolving;completion;neutralized;

- ure of compound 274 (6.4 g, 17.2 mmol), iron powder (2.89 g, 51.8 mmol) and ammonium chloride (4.61 g, 86.2 mmol) in MeOH (100 ml) and H.sub.2 O (100 ml) was heated to reflux for 4 h. The reaction mixture was filtered while hot through celite and the aqueous layer extracted with EtOAc. The **organic** layer was washed with brine, dried and evaporated. The **residue** was dissolved in CH.sub.3 CN (100 ml) and **acetic acid** (1 ml) and heated to reflux 4 h. The solvent was removed and the **residue** purified by flash chromatography eluting with hexanes-EtOAc (2:1 to 1:2) to yield 4.17 g (75%) of compound 274a as a viscous oil.
 .sup.1 H NMR (CDCl.sub.3) .delta.1.86-1.98 (m, 2 H), 2.38-2.51 (m, 2 H), 3.34-3.39 (m, 1 H), 3.80-3.87 (m, 2 H), 4.06-4.14 (m, 1 H), 4.38-4.66 (m, 2 H), 7.18-7.19 (m, 1 H), 7.26-7.40 (m, 6 H), 7.72-7.74 (m, 1 H); MS m/e 372 (MH.sup.+).
 ##STR476##

To a solution of compound 274a (3.23 g, 10 mmol) in CH.sub.2 Cl.sub.2 at

----- 4350606
 classes:1 252/392 1 106/1415 1 106/1416 1 106/1442 1 252/364 1 422/16 1 510/255 1 510/266 1 510/499 1 516/
 67 1 516/68 1 516/7 1 564/300 1 564/503
 score: 351

keywords: water;N-methyl;alkyl;claimed;hydroxylamine;ratio;solution;compounds;amine;aqueous;metal;amines;
 novel;dioxide;adding;water-soluble;oxygen;

----- 6458849
 classes:1 514/649 1 514/654 1 514/655 1 514/658 1 564/374 1 564/381 1 564/382 1 564/384 1 564/389 1 564/3
 90 1 564/391 1 564/392 1 564/440

score: 337

keywords: water;dms;dimethylsulfoxide;acetic acid;removing;polyethylene glycol;polyethylene;N-methyl;carbonate;propylene glycol;propylene;ethylene glycol;ethyl acetate;acetic acid;acetic;hydroxide;aqueous solution;ethanol;alkyl;organic;fluoride;residue;corresponding;sulfone;independently;dimethylsulfoxide;hydroxylamine;hydrazine;ratio;solution;hydrogen;compounds;solvent;amine;ammonium;aqueous;metal;acid;methyl;acetate;ethyl;ethylene;glycol;concentrations;carboxylic;ketone;beneficial;nitrogen;carbonyl;hydroxyl;functional groups;substituted;ingredients;blend;ambient;Stirring;derivatives;magnesium;benzoic;organic acids;hydrochloric acid;hydrochloric;phosphoric;sulfuric;zinc;potassium;amides;listed;dimethyl;desirably;discloses;ammonia;amines;inhibit;novel;adding;fluorinated;solubility;European;published;quaternary ammonium;quaternary;ketones;phase;issued;formulation;temperatures;formulations;hydrocarbon;aromatic;phenolic;dissolving;completion;acidic;neutralized;aluminum;oxygen;reacted;